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14. ABSTRACT

Organophosphorous (OP) cholinesterase inhibitors remain a significant threat to military and civilian personnel. Reactivators of OP inhibited cholinesterases can serve as OP agent antidotes but can be limited by their poor bioavailability and poor reactivation kinetics. New reactivator designs may be able to address these shortcomings. This proposal explores and evaluates the feasibly of synthesizing two new classes of oxime reactivators that use bifunctional catalysis to enhance kinetics and improve bioavailability. The synthesis of amino-functionalized aryltrifulorketoxmines and 3-alkylaminopyridinium oximes are described.

15. SUBJECT TERMS
Cholinesterase Reactivator, Synthesis, Oxime, acetylcholinesterase

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Table of Contents

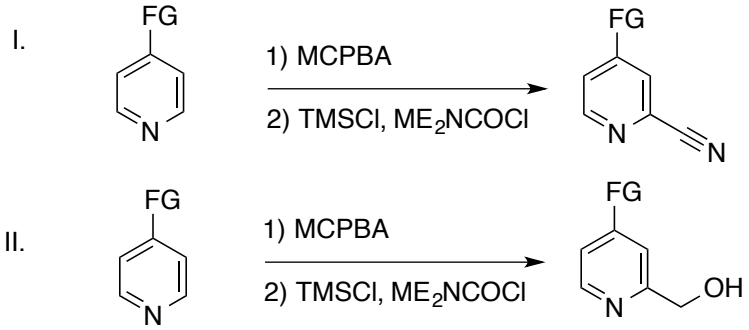
INTRODUCTION:

This objective of this project is to explore the synthetic feasibility to construct bifunctional oxime reactivators that bear pendant amine or related basic functionality. Basic functional groups have the potential to enhance the kinetic rate of oxime reactivators through participation in general acid catalysis. Three general classes of compounds have been devised in the statement of work. (I.) amine functionalized pyridinium oximes, (II) Electron deficient pyridine and benzaldoximes and (III) bifunctional analogs of MINA.

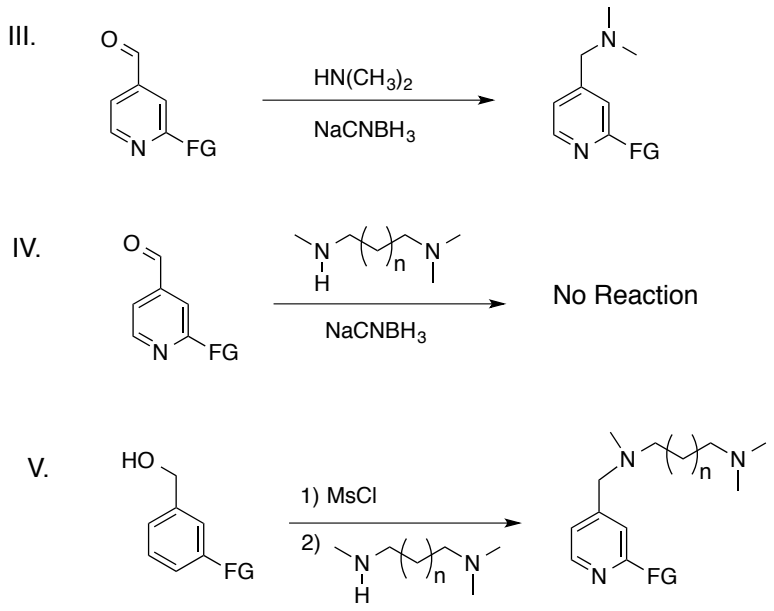
BODY:

We reduced to practice a general method for making novel bifunctional oxime AChEreactivators based on 2-PAM designed to increase the rate-limiting step of reactivation using substrate assisted catalysis. One of the key proposed strategies was to develop an approach to 2-pyridinium oximes that contains amine or similar functionality tethered to the 4-position that could act in general acid catalysis.

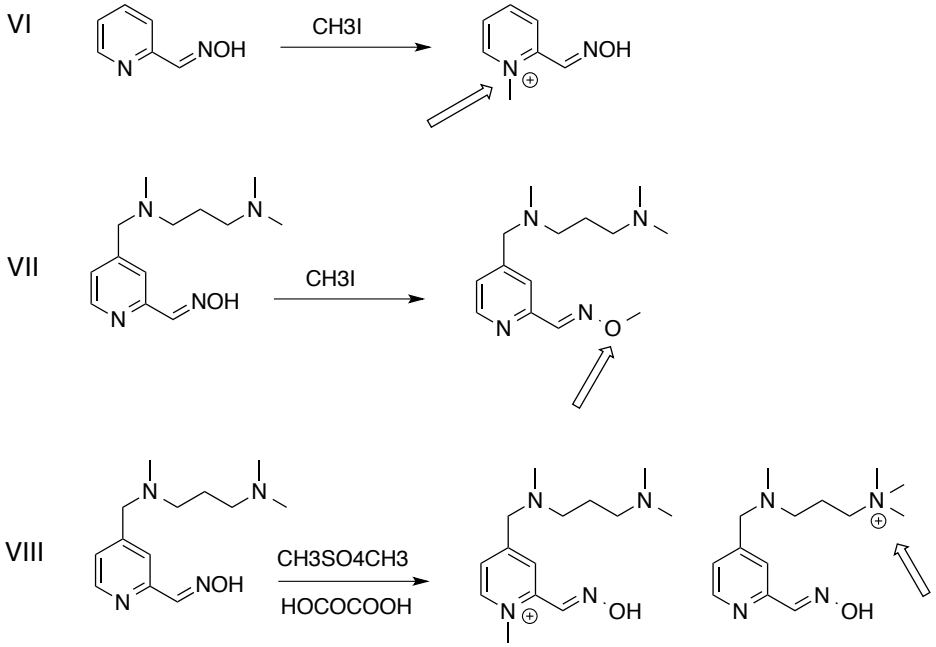
General considerations for constructing 2-Pyridinium oximes: Two general approaches were explored for the synthesis of bifunctional oximes, One strategy (I), was introduce the 2-aldehyde as a nitrile via the pyridine N-oxide. Another approach (II), was to simply perform 2-hydoxymethylation of pyridines by radical addition using ammonium persulfate (APS). Although both strategies were effective, selective reduction of the nitrile proved difficult in the presence of amide and diamine functionalities. With the availability of commercially available hydoxymethyl pyridines, the latter approach was generally more practical except for the synthesis of amidines and amidoximes (See below).



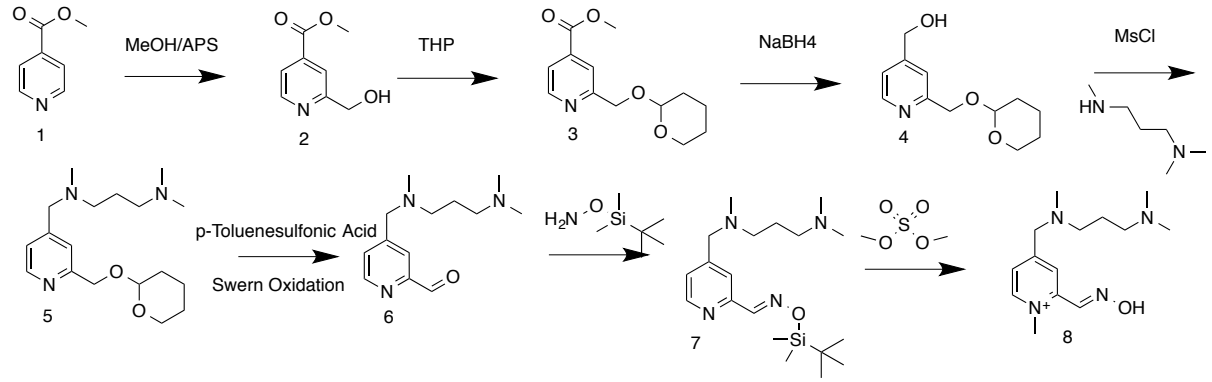
A key compound was to develop 2-PAM analogs with amine and or diamine functionality in the 4 positions. Our original design plan called for introducing an aldehyde that could be coupled to various amines and diamines through reductive amination. Interestingly while amines readily coupled under these conditions, the diamines, called for by our design, did not couple under these conditions. Search of the literature revealed a dearth of successful exmples of reductive ammination by



Our revised strategy for construction of 4 substituted bifunctional pyridinium oximes is illustrated in Scheme 1. The resulting strategy is a little longer than our initial design but provides flexibility for a generalizable scheme. A key finding from this approach has been that the oxime, which normally does not need protection in order to perform selective N-methylation of the pyridine nitrogen in the synthesis of 2-PAM, is preferentially methylated in the presence of the diamine (VI). Presumably the strongly basic diamine deprotonates the oxime under standard methylation conditions.



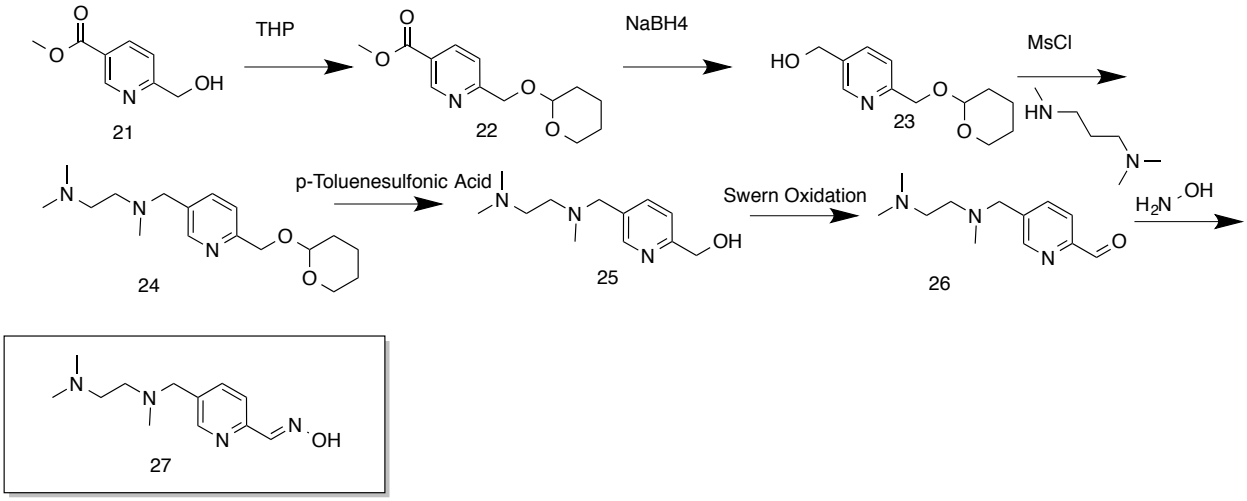
Protection of the oxime as theTBDMS (tButyldimethylsilyl) ether blocks O-alkylation (VII). The diamine alkylation is still competitive for N-pyridine alkylation but can be controlled by "buffering" the reaction such that the more basic amines are protonated (VIII).



Scheme 1. The synthesis of oxime 8 illustrates a general strategy for constructing bifunctinoal oxime reactivators with pendant general acid groups.

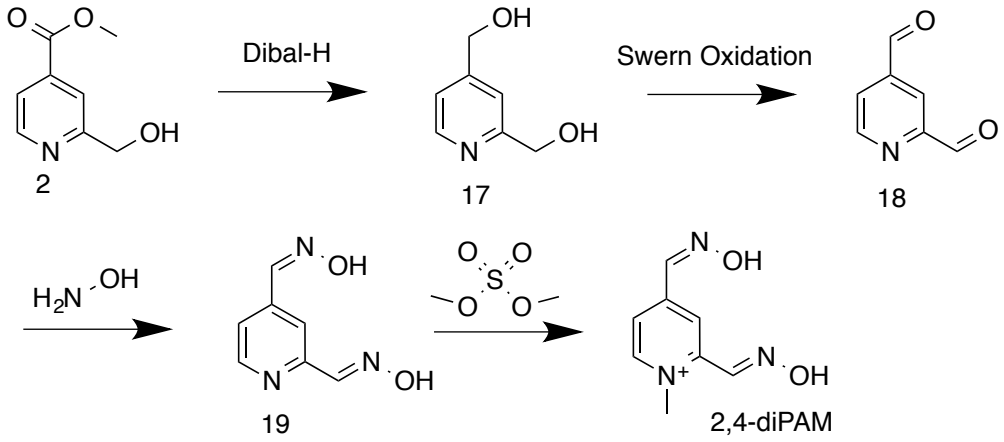
Compound 8 has been delivered to ICD for testing (synthesis and characterization below).

This strategy is in place to make additional analogs such as the 5-substituted analog **27**.



Scheme 2. Synthesis 5-substituted analogs of 2-PAM with pendant diamine.

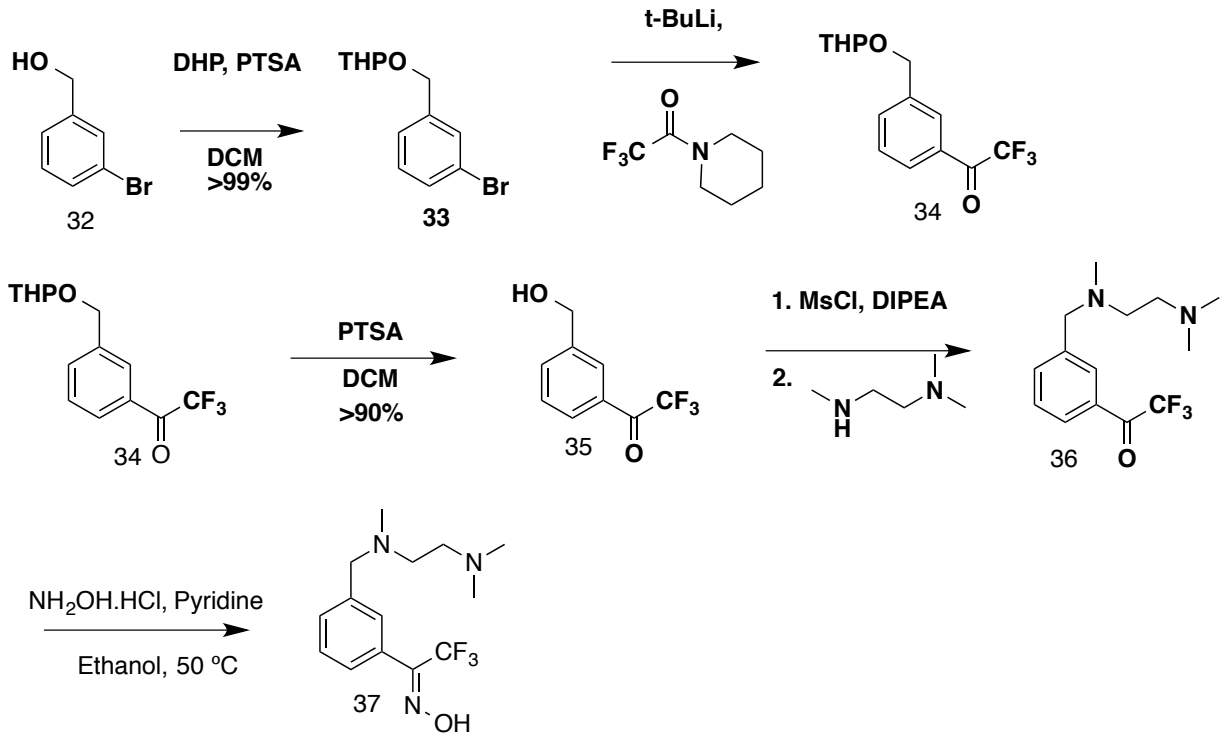
As a reference compound we also synthesized 2,4-diPAM.



Scheme 3. Synthesis of 2,4DiPAM

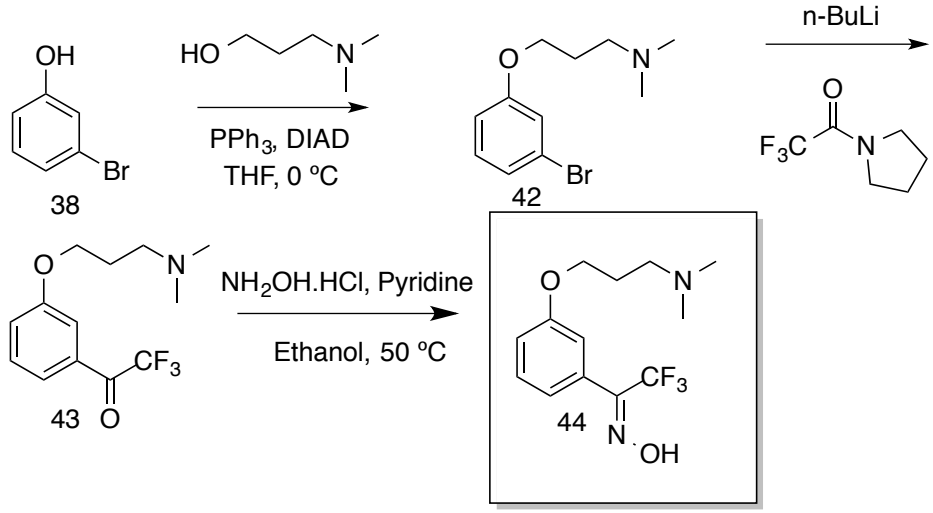
As part of objective 2 as defined in the SOW, we will assess the synthetic feasibility of making electron deficient bifunctional benzaldoximes and benketoximes and related hydroxamic acids and amidoximes as potential bioavailable AChE reactivators capable of substrate-assisted catalysis. We have explored way to synthesize non-cationic analogs of 2-PAM that can be used to lower the oxime pKa to facilitate catalysis without using a permanent charge.

Trifluoromethylketoximes: We developed a general route to the synthesis of trifluoromethylketoximes. Compound **37** was made as an isostere of the 4-substituted bifunctional oximes. Starting from 3-bromobenzylalcohol, the alcohol was protected as a THP ether and the trifluoromethylketone installed by coupling the organolithium with trifluoroacetylpiperidine. We again used our direct acylation strategy to install the diamine side chain, which was believed to provide an amine cation for AChEbinding/recognition. In vitro testing suggests that these compounds function as inhibitors. We later speculated that the trifluoro-methylketoxime may be might be forming covalent adducts through haloform chemistry.

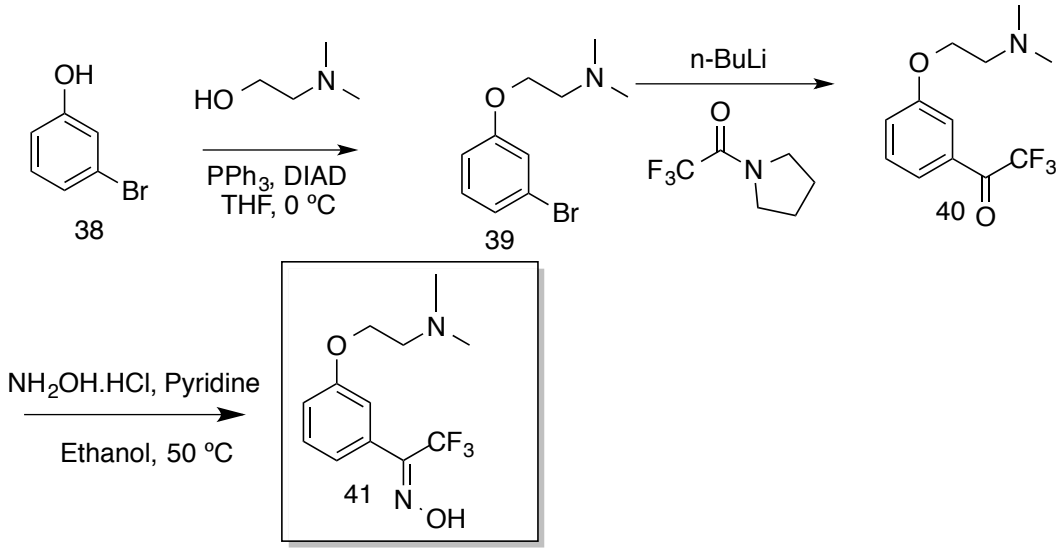


Scheme 4.. Synthesis of bifunctional trifluoroketoxime **47.**

To further modulate the trifuoromethylketoxime pKa we also synthesized analogs with 4-aminoalkoxy substituents. The alkoxy substitution should be electron donating and raise the effective pKa of the oxime. These could be made from Mitsinobu coupling of 3-bromophenol. These analogs **41** and **44** were synthesized, characterized and delivered to ICD.

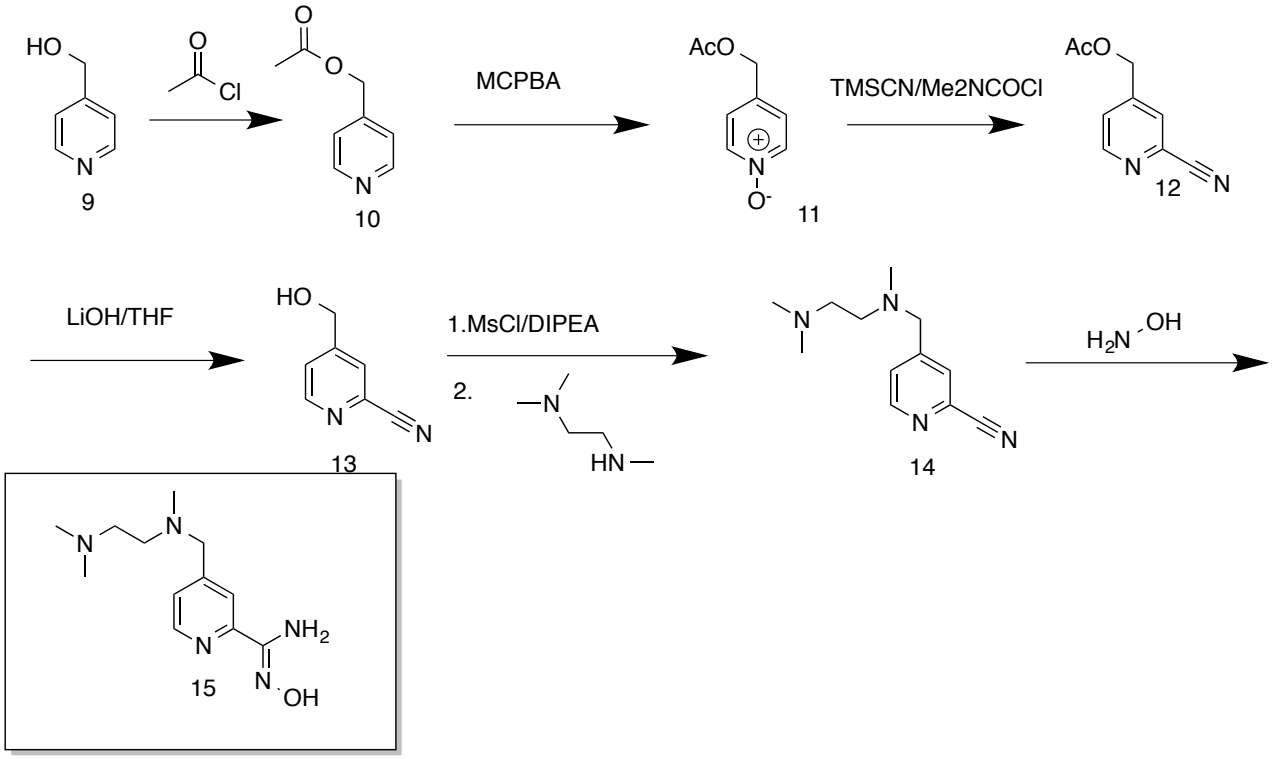


Scheme 5. Synthesis of trifluoroketoxime **47.**



Scheme 6. Synthesis of trifluoroketoxime **41.**

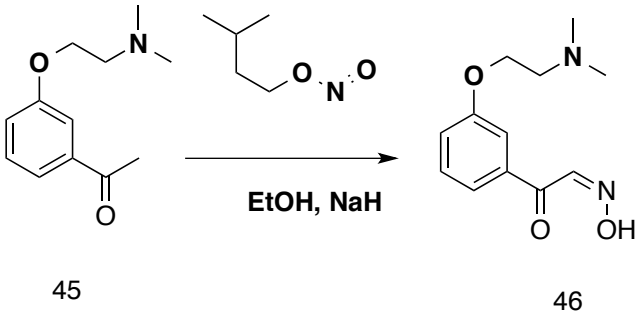
Amideoxime: Another oxime analog we explored was the amideoxime. The amideoxime **15** was made by direct addition of hydroxylamine to the nitrile **14** which was made following the route initially described to developed to bifunctional pyridinium oximes.



Scheme 7. Synthesis of bifunctional amidoxime analog.

MINA and RS41 analogs: We have made initial progress towards the synthesis of MINA/RS41A analogs. The remainder of the project period, expanded through no-cost extension) will be focused on making additional MINA analogs.

We have made the novel amine-functionalized nitrosoacetophenone **46** through direct nitrosylation of the acetophenone.



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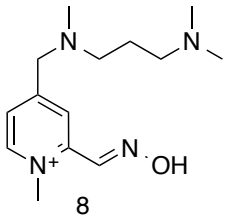
Scheme 8. Synthesis of α-nitrosobenzophenone analog.

We have reduced to practice a general strategy for synthesizing a novel MINA analog that uses an amine functionalized acetophenone core.

KEY RESEARCH ACCOMPLISHMENTS:

SOW objective 1: We have successfully demonstrated the synthetic feasibility of synthesizing bifunctional pyridinium oximes.



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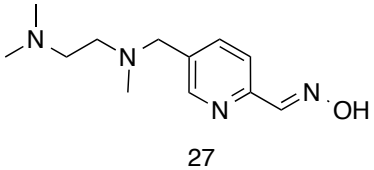
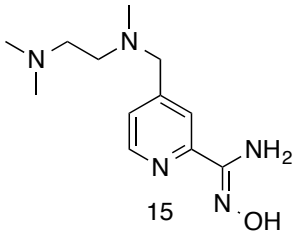


27

- General strategies for synthesizing 4-aminoalkyl substituted pyridinium oximes has been achieved
- General method for 5-aminoalkyl substituted pyridine oxime has been achieved.

SOW objective 2: We have successfully demonstrated the synthetic feasibility of constructing electron deficient bifunctional benzaldoxmine, ketoximes and amidoximes.



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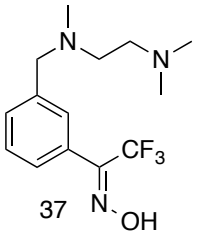

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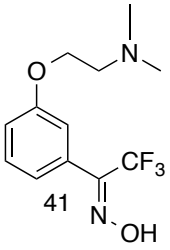

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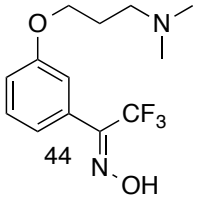
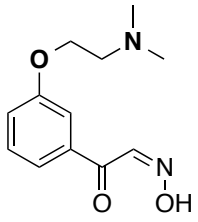


44

- 4-aminoalkyl substituted pyridine amideoximes have been synthesized
- 4-aminoalkyl substituted trifluoroacetophenone oximes have been synthesized
- 4-aminioalkyl substituted trifluoroacetophenone oximes have been synthesized

SOW objective 3. Assess the synthetic feasibility of making MINA and RS41A analogs designed to support a substrate assisted bifunctional catalytic mechanism



46

REPORTABLE OUTCOMES:

None

CONCLUSION: We have successfully evaluated the synthetic feasibility of bifuncional pyridinium oximes and electron deficient oxime analogs as outlined in objectives on SOW objectives 1 and 2. This work has resulting in the development of a general scheme for oxime protection and for selective pyridine N-alkylation by adjusting the pH to reduce/prevent side chain alkylation. We have successfully made 7 new reactivator analogs that incorporate new functionalities that are in principle capable of supporting general acid/base catalysis. Preliminary studies suggest that trifluoroacetophenoximes are inhibitors of AChE.

In this report period work has begun on objective 3, evaluating the synthetic feasibility of constructing bifunctional MINA analogs. We hope to complete the synthesis of new MINA analogs described in objective 3 and additionally complete new pyridininum based oxime analogs.

APPENDICES:

General Procedures:

Synthesis of **2**: To a solution of **1** (10g, 72.92 mmol) and H_2SO4 in MeOH under refluxing was added a solution of APS (30g, 131.256 mmol) in water over 20 minutes. The solution was allowed to cool to room temperature. The MeOH was removed and solution was neutralized by Na_2CO_3 to PH as 8. The aqueous layer was extracted by EtOAc, combined and purified by flash column chromatography (EtOAc : Hex = 1 : 1).

Synthesis of **3**: To a solution of **2** (1g, 6.5 mmol) in DCM was added p-tolunesulfonic acid (25mg, 0,1318 mmol), followed by addition of dihydropyran, stirred at RT for 2 hours, after which the reaction was poured into $NaHCO_3$ and extracted by EtOAc. The solvent was removed by reduced pressure and the residue was purified by flash chromatography with EtOAc : DCM = 1: 2.

Synthesis of **4**: To a solution of **3** (588.3mg, 2.344mmol) in 50 ml EtOH was slowly added $NaBH_4$ (443mg, 11.72 mmol)and than heated to reflux for 2 hours. The reaction was quenched by adding water, the EtOH was removed by reduced pressure, and aqueous layer was extracted with DCM. The residue was purified by flash chromatography with MeOH : DCM = 1 : 19.

Synthesis of **5**: To a solution of **4** (152mg, 0.68 mmol) in DCM in a ice bath was added DIPEA (548 uL, 3.151 mmol) than MsCl (160 uL, 2.055 mmol), stirred for 75 minutes, after which the solution was added drop-wisely to a solution of ethylenediamine in DCM in a ice bath. The reaction was stirred for another one hour, the solvent was removed by reduced pressure and the residue was purified by flash chromatography $MeOH.NH_3$: DCM = 1 : 19.

Synthesis of **6**: To a solution of **5** in MeOH was added TSA in a ice bath. The reaction was stirred for 16 hours, concentrated and redissolved in $CHCl_3$, washed with $NaHCO_3$ and dried. The aldyhyde intermediate was applied to next step without further purification.

Synthesis of **7**: To a solution of **6** (.2273 mmol) in 5 ml of EtOH was added TBDMS hydroxyamine (147 mg, 100 mmol) and 55 uL of pyridine. The reaction was heated to 40^{o}C for 4 hours, after which the solvent was removed by reduced pressure and the residue was purified by flash chromatography NH_3.MeOH : DCM = 1 : 19.

Synthesis of **8**: To a solution of **7** (11.85 mg, 0.034 mmol) in EtOH was added oxalic acid and reflux for 15 minutes, after which the solvent was removed by reduced pressure. To the residue was added THF and Dimethyl sulfate (6.42 uL, 0.068 mmol). The reaction was stirred at 50^{o}C for over night. The precipitate was applied to reverse phase column with H_2O : MeOH = 19 : 1

Synthesis of **10**: To a solution of **9**, 4-pyridyl carbinol (1g 9.16 mmol) and TFA (1.23 mL, 16.5 mmol) in 4 mL toluene was added a solution of acetyl chloride (1.05 mL, 14.7 mmol) in DCM drop wise. The reaction was stirred for 10 hours, after which water was added to the reaction and organic layer was dried and concentrated in vacuo. The residue was purified by flash chromatography with MeOH : DCM = 1 : 19.

Synthesis of **11**: To a solution of **10** (700 mg, 4.64 mmol) in 4 mL acetone was added a solution of m-CPBA (1.37 g, 5.56 mmol) in 4 mL acetone drop wise in 4 minutes. The reaction was stirred for 4 hours, after which the solvent was removed by reduced pressure and the residue was portioned between water and ether. The organic layer was dried and concentrated in vacuo. The residue was purified by flash chromatography with MeOH : DCM = 1 : 19.

Synthesis of **12**: To a solution of **11** (162.4 mg, 0.97 mmol) in DCM was added TMSCN (145 uL, 1.16 mmol), after which Me_2NCOCl (106.8 uL, 1.16 mmol) was added in three portions over thirty minutes. The reaction was stirred for over night, then the solvent was removed by reduced pressure and the residue was purified by flash chromatography with EtOAC : Hexane = 3 : 7.

Synthesis of **13**: To a solution of **12** (360 mg, 2.04 mmol) in 10 ml THF was added 1N LiOH (4.1 mL). The reaction was stirred for 2 hours. THF was removed by reduced pressure and the aqueous layer was exhaustively extracted by DCM. The organic layers were combined and concentrated by vacuo. The residue was applied to next step without purification.

Synthesis of **14**: To a solution of 13 (184 mg, 1.37 mmol) in 8 mL DCM was added DIPEA (550 uL, 3.151 mmol) and MsCl (122 uL, 1.58 mmol) in a ice bath. The reaction was stirred for 75 minutes and then was added to a solution of trimethylethenediamine (500 uL, 3.425 mmol) in DCM, than warmed to RT. The reaction was concentrated in vacuo and the residue was purified by flash chromatography.

Synthesis of **15**: To a solution of **14** (85mg, 0.39 mmol) in 6 mL EtOH was added Hydroxylamine and pyridine. The reaction was refluxed for 4 hours, than stirred at RT for over night. The reaction was concentrated in vacuo and the residue was purified by flash chromatography with NH_3.MeOH : DCM = 1 : 9

Synthesis of **17**: To a solution of **2** (347 mg, 2.065 mmol) in THF was added Dibal-H (6.2 ml, 6.2 mmol) drop wisely at -78^oC for 3 hours. The reaction was quenched by addition of Sodium Sulfate Decahydrate and stirred for over night. The solvent was removed in vacuo and the residue was purified by flash chromatography Meoh : DCM = 1 : 19.

Synthesis of **18**: Oxalyl Chloride (55 uL, 0.64 mmol) in DCM was cooled to -78^oC, into which was added DMSO (90.1 uL, 1.28 mmol) drop wisely, in 10 minutes, solution of **17** (22.3 mg, 0.16 mmol) in DCM was added to the solution. The reaction was stirred at -78^oC for 45 minutes before DIPEA (45 uL, 2.56 mmol) was added and the reaction was warmed up to 0^oC. The reaction was poured into water and was extracted with DCM. The organic layers were combined and concentrated in vacuo. The product was applied to next step without further purification.

Synthesis of **19**: To a solution of **18** (51.4 mg, 0.38 mmol) in 5 mL EtOH was added Hydroxylamine hydrochloride. The reaction was refulxing for 4 hours, than at RT for overnight. The solvent was removed by reduced pressure and the residue was purified by flash chromatography with MeOH : DCM = 1 : 19.

Synthesis of **20**: To a solution of **19** (26.8 mg, 0.1624 mmol) in anhydrous THF was added Dimethyl Sulfate (16 uL, 0.1624 mmol) at RT, the reaction was stirred for over night. The solvent was taken out by a pippett and the precipitate was purified by a reverse column, with MeOH : H_2O = 1 : 19.

Synthesis of **22**: To a solution of **21** (1.24g, 7.54 mmol) in 50 mL DCM was added p-toluenesulfonic acid , followed by addition of dihydropyran. The reaction was stirred at RT for w hours, after which the reaction was poured to Saturate $NaHCO_3$ and extracted by EtOAC. The organic layer was dried and concentrated in vacuo. The residue was purified by flash chromatography with EtOAC : DCM = 1: 9.

Synthesis of **23**: $NaBH_4$ (567.3 mg, 15 mmol) was slowly added to a solution of 22 in EtOH at RT, than the reaction was heated to reflux for 2 hours. The solution was cooled to RT and water was added. The solution was concentrated and basified by $NaHCO_3$ and extracted by DCM. The solution was concentrated in vacuo and the residue was purified by flash chromatography with

MeOH : DCM = 1 : 19.

Synthesis of 24: To a solution of **23** (147 mg, 0.6632 mmol) in DCM in a ice bath was added DIPEA (265 uL, 0.99 mmol) and MsCL (76 uL, 0.99 mmol), in 75 minutes, the solution was added drop wisely to a solution of trimethylehylenedimaine (194 uL, 1.3264 mmol) in a ice bath and warm up to RT. The solution was concentrated in vacuo and purified by flash chromatography with NH_3.MeOH : DCM = 1 : 9.

Synthesis of **25**: To a solution of 24 (84 mg, 0.274 mmol) in MeOH was added p-toluenesulfonic acid (208.2mg, 1.09 mmol) in a ice bath. The reaction was stirred for 16 hours, concentrated, redissolved in chloroform. The solution was washed with minimum amount of saturate $NaHCO_3$ and dried purified by flash chromatography with NH_3.MeOH : DCM = 1 : 9.

Synthesis of **26**: A solution of Oxalyl chloride (55.45 uL, 0.646 mmol) in 2 mlL of DCM was cooled to -78^{o}C and was added to a solution of DMSO (91.6 uL, 100.79 mmol) drop wisely. The reaction was stirred for 10 minutes, after which a solution of 25 (72 mg, 0.323 mmol) in DCM was added over 10 minutes. The mixture was stirred at -78^{o}C for 45 minutes before DIPEA (451 uL, 2.59 mmol) was added swiftly than the slurry was warmed up to 0^{o}C and stirred for another 30 minutes. Than the reaction was poured to water and extracted by DCM. The solvent was removed by reduced pressure and purified by flash chromatography with MeOH : DCM = 1 : 9

Synthesis of **27**:To a solution of 26 (71 mg, 0.323 mmol) in EtOH was added H_2NOH.HCl (67.35 mg, 0.969 mmol) and pyridine (78 uL, 0.969 mmol). The reaction was heated to 40^{o}C for one hour, than lowered to RT, stirred for over night. Than the solvent was removed by reduced pressure and the residue was purified by flash chromatography with NH_3.MeOH : DCM = 1 : 19.

Synthesis of **33**: 3-bromobenzylalcohol(10g, 53.46 mmol) in DCM was treated with DHP(6 mL, 64.15mmol) and dry p-toluenesulfonic acid (100 mg). The reaction was stirred overnight. The reaction was extracted with H_2O and DCM. The aqueous layer was extracted 3 × 100 mL DCM and the combined organic layer was washed with 3 × 50 mL brine, dried over MgSO4 and concentrated in vaccuo. The product was separated using column chromatography Hexane: Ethylacetate (90:10) as a white solid.

Synthesis of **34** (trifluoroacetylation general procedure.)
To 33(2.556 g, 9.42 mmol) in THF at -78 ºC was added n-BuLi (14.81 mL, 20.74 mmol). The reaction was stirred at -78 ºC for 30 mins. Following which 2,2,2-trifluoro-1-(pyrrolidin-1-yl)ethanone (1.23 mL, 10.362 mmol)was added dropwise. The reaction was stirred at -78 ºC for 1h and allowed to warm to room temperature. Following which the reaction was worked up with saturated NH4Cl and ether. The aqueous layer was extracted with 50 mL ether. The combined organic layer was washed with brine, dried over MgSO4 and concentrated in vaccuo.

Synthesis of **35** (THP deprotection common procedure)
To 2,2,2-trifluoro-1-(3-(((tetrahydro-2H-pyran-2-yl)oxy)methyl)phenyl)ethanone (768.5 mg, 2.66 mmol) in DCM was added dry p-toluenesulfonic acid (50.71 mg, 1900.22 mmol) and the reaction was stirred at room temperature for 1 h. The reaction was stirred overnight. The reaction was extracted with H2O and DCM. The aqueous layer was extracted 3 × 100 mL DCM and the combined organic layer was washed with 3 × 50 mL brine, dried over MgSO4 and concentrated in vaccuo. The product was separated using column chromatography Hexane: Ethylacetate (90:10) as a white solid.

Synthesis of **36** (trimethyldiaminne displacement common procedure): To 2,2,2-trifluoro-1-(3-(hydroxymethyl)phenyl)ethanone (225.8 mg, 1.106 mmol) in DCM was added

Methansulfonylchloride (100 uL, 139.42 mmol) and DIPEA (423 uL, 314.44 mmol). The reaction was stirred at room temperature for 1 h. The reaction mixture was worked up with H2O and DCM. The reaction was extracted with H2O and DCM. The aqueous layer was extracted 3 × 50 mL DCM and the combined organic layer was washed with 3 × 30 mL brine, dried over MgSO4 and concentrated in vaccuo. The crude product in 10 mL DCM was treated with N,N,N'-trimethylethane-1,2-diamine and reaction stirred overnight. The reaction was extracted with H2O and DCM. The aqueous layer was extracted 3 × 100 mL DCM and the combined organic layer was washed with 3 × 50 mL brine, dried over MgSO4 and concentrated in vaccuo. The product was separated using column chromatography DCM: MeOH (90:10) as a yellow oil.

Synthesis of **37** (Oxime formation general procedure)
To the ketone **36** (340 mg, 1,123 mmol) in 1 ml of pyridine was added Hydroxylamine hydrochloride (312.15 mg, 4.492 mmol). The reaction was stirred overnight at 50 ºC. After which pyridine was evaporated and the residue was applied on the silica column and product was separated using DCM:MeOH (NH3).

Synthesis of **39** (Mitsunobo reaction general procedure): To 3-bromophenol and triphenylphosphine in THF at 0 ºC was added 2-dimethylaminoethanol. To this solution was added diisopropylazidodicarboxylate. The mixture was allowed to stir at room temperature overnight. The reaction was worked up with H2O and the aqueous layer was extracted with ether. The organic layer was washed with 3 brine, dried over MgSO4 and concentrated in vaccuo. the residue was dissolved in minimum amount of hexane and cooled at -20 ºC to crystalize triphenyl phosphineoxide. The supernatant was applied on to silica column to separate the product using DCM:MeOH.

Synthesis of **40** (Trifluoroacetylation general procedure): To 2-(3-bromophenoxy)-N,N-dimethylethanamine (683.8 mg, 2.8 mmol) in THF at -78 ºC was added n-BuLi (5 mL, 7 mmol). The reaction was stirred at -78 ºC for 30 mins. Following which 2,2,2-trifluoro-1-(pyrrolidin-1-yl)ethanone (668 uL, 795.31 mmol) was added dropwise. The reaction was stirred at -78 ºC for 1h and allowed to warm to room temperature. Following which the reaction was worked up with saturated NH4Cl and ether. The aqueous layer was extracted with 50 mL ether. The combined organic layer was washed with brine, dried over MgSO4 and concentrated in vaccuo.

Synthesis of **41**: Oxime **41** was made from ketone 40 following the same proceed common procedure used to make compound **37**.

Synthesis of 42: Ether **42** was made from 3-bromophenol by Mitsunobo reaction following the same general procedure was applied to compound **38**.

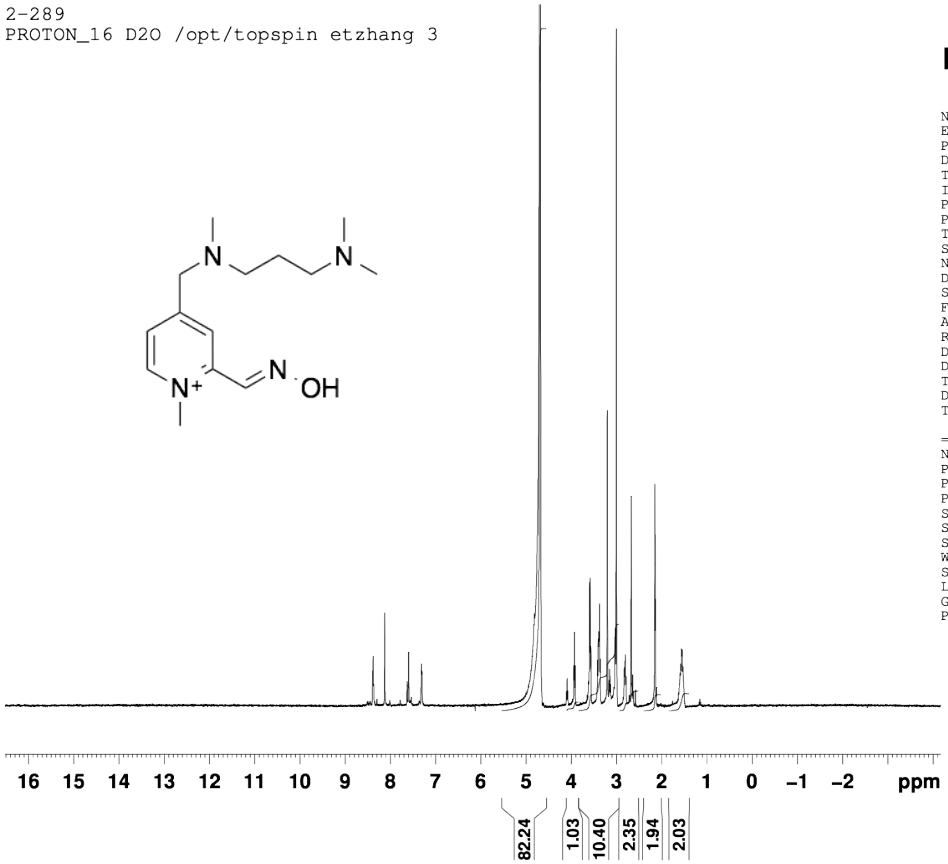
Synthesis of **43**: Compound **43** was made by trifuloroacetylation of compound **42** following the same general procedure used to make compound **40**.

Synthesis of **44**: Oxime **44** was made from ketone **43** using the same general procedure used to make compound **37**.

Synthesis of 45
To 5ml of Ethanol was added NaH (75 mg, 3.12 mmol) in small portions to generate sodiumethoxide. To a solution of 1-(3-(2-(dimethylamino)ethoxy)phenyl)ethan-1-one (534.3 mg, 2.6 mmol) in 500 uL EtOH was added the freshly made Sodium ethoxide dropwise. To this mixture was added dropwise isoamylnitrite (335.04 mg, 2.86 mmol). The mixture was stirred overnight. The solvent was evaporated and the product separated on reverse phase c-18 column using H2O:MeOH (90:10). The solvent was evaporated and residual water was removed by

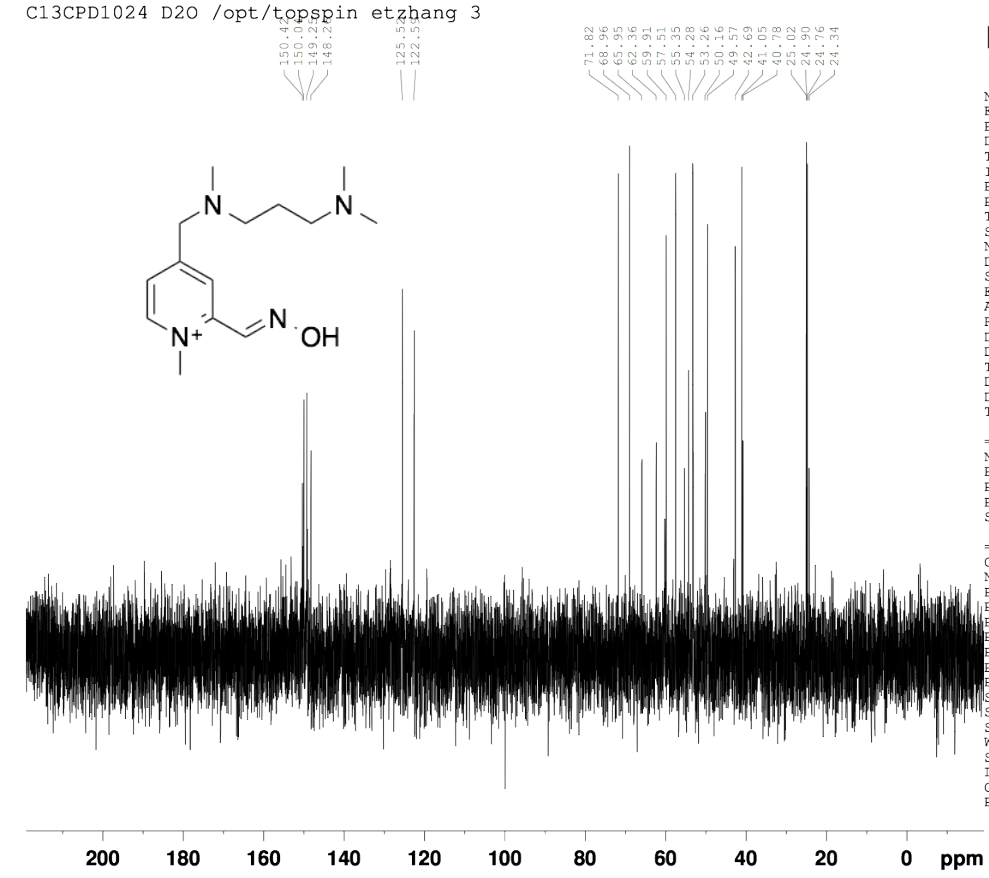
lyophylization. to yield a yellow solid.

2-289
PROTON_16 D2O /opt/topspin etzhang 3



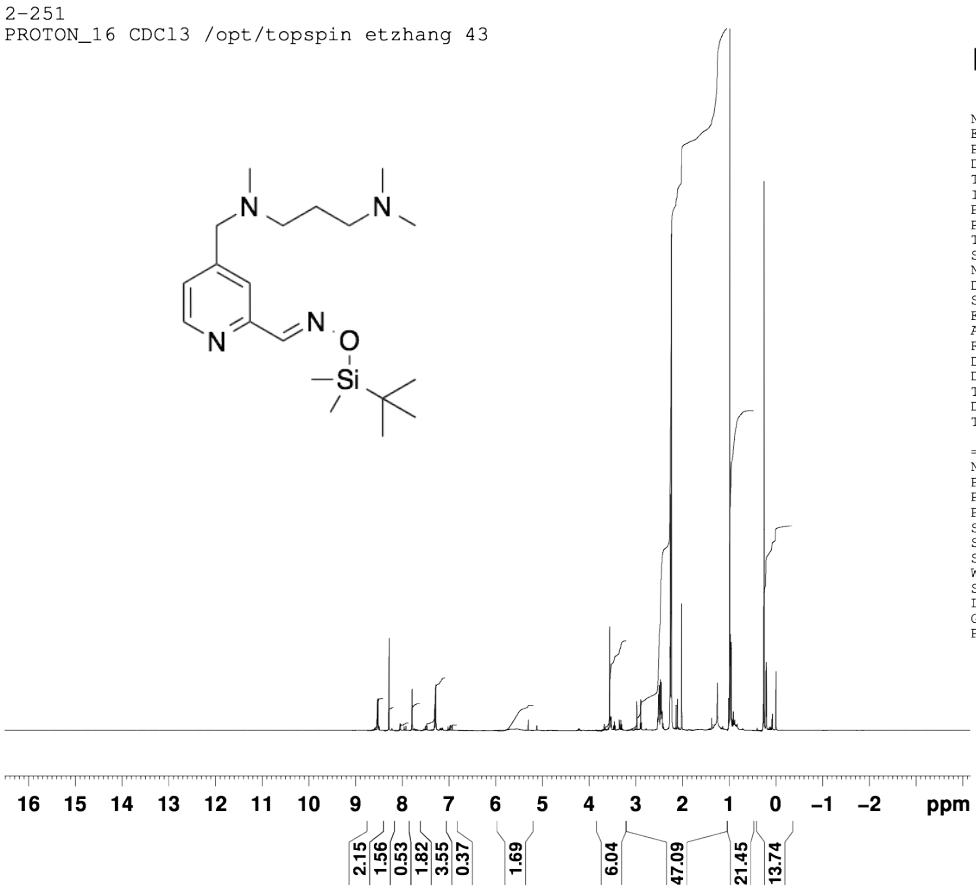
NAME	2-289
EXPNO	1
PROCNO	1
Date_	20130524
Time	17.24
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zg30
TD	65536
SOLVENT	D2O
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	9
DW	60.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

2-289
C13CPD1024 D2O /opt/topspin etzhang 3



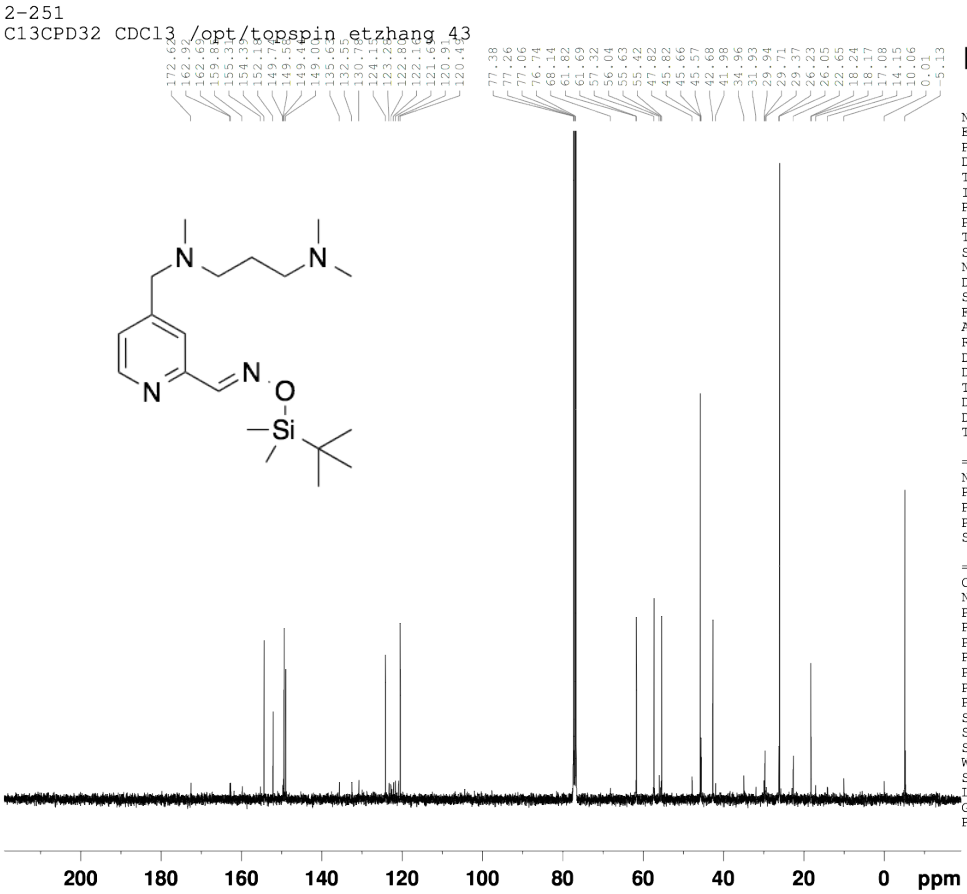
NAME	2-289
EXPNO	4
PROCNO	1
Date_	20130525
Time	3.14
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	D2O
NS	1024
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	512
DW	20.850 usec
DE	18.00 usec
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.25 usec
PL1	0.55 dB
PL1W	35.18820572 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	4.90 dB
PL12	20.46 dB
PL13	21.00 dB
PL2W	3.30822015 W
PL12W	0.09195905 W
PL13W	0.08120718 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

2-251
PROTON_16 CDCl3 /opt/topspin etzhang 43



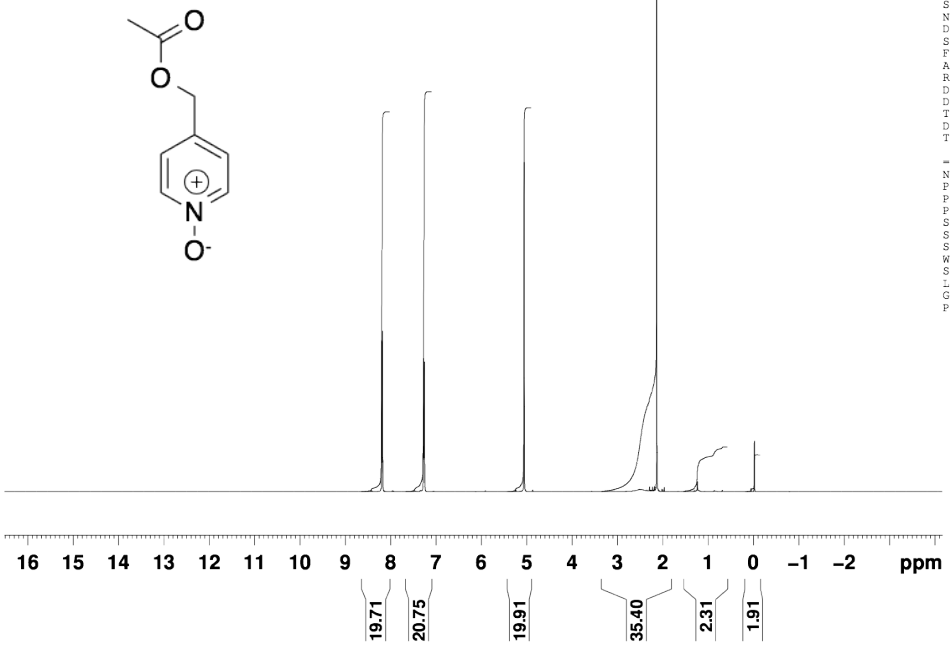

```
NAME                2-251
EXPNO                   1
PROCNO                  1
Date_            20130521
Time                23.49
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8278.146 Hz
FIDRES           0.126314 Hz
AQ               3.9584243 sec
RG                    6.3
DW                 60.400 usec
DE                  6.00 usec
TE                 298.1 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  15.00 usec
PL1                  4.90 dB
PL1W          3.30822015 W
SFO1         400.1324710 MHz
SI                  32768
SF           400.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

2-251
C13CPD32 CDCl3 /opt/topspin etzhang 43



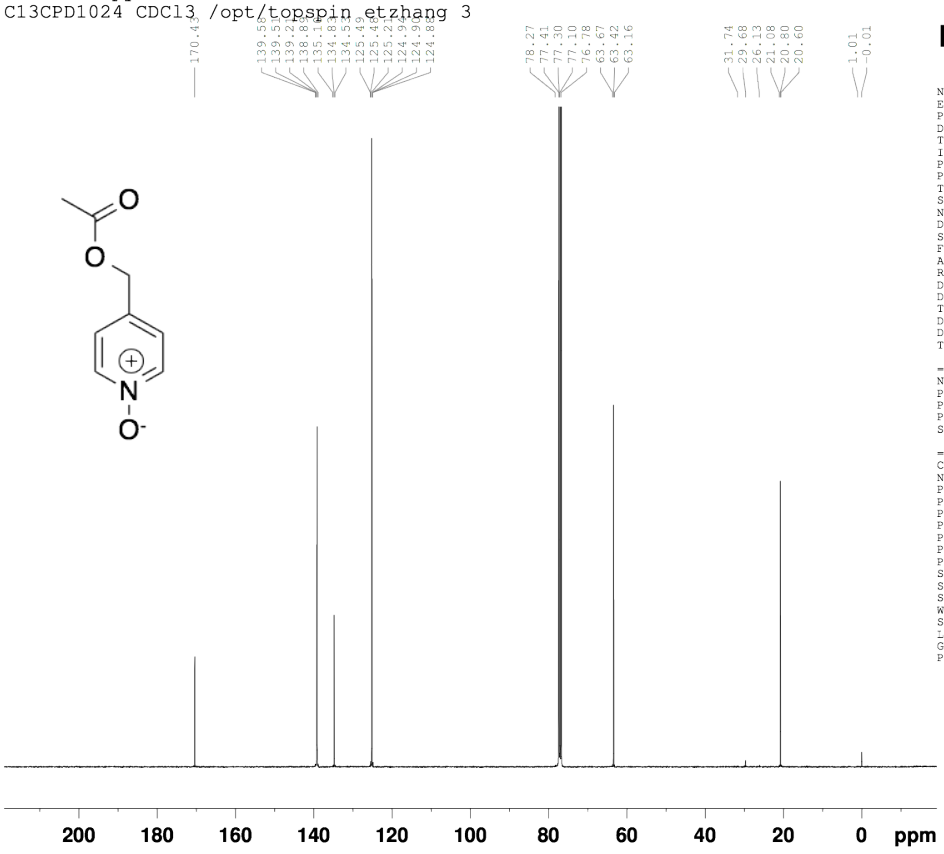

```
NAME                2-251
EXPNO                   3
PROCNO                  1
Date_            20130522
Time                 0.05
INSTRUM             spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH             23980.814 Hz
FIDRES           0.365918 Hz
AQ               1.3664756 sec
RG                    512
DW                 20.850 usec
DE                  18.00 usec
TE                 298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.25 usec
PL1                  0.55 dB
PL1W         35.18820572 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                  4.90 dB
PL12                20.46 dB
PL13                21.00 dB
PL2W          3.30822015 W
PL12W         0.09195905 W
PL13W         0.08120718 W
SFO2        400.1316005 MHz
SI                  32768
SF          100.6127690 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

2-102-ac-pyridine-n-oxide
PROTON_16 CDCl3 /opt/topspin etzhang 3



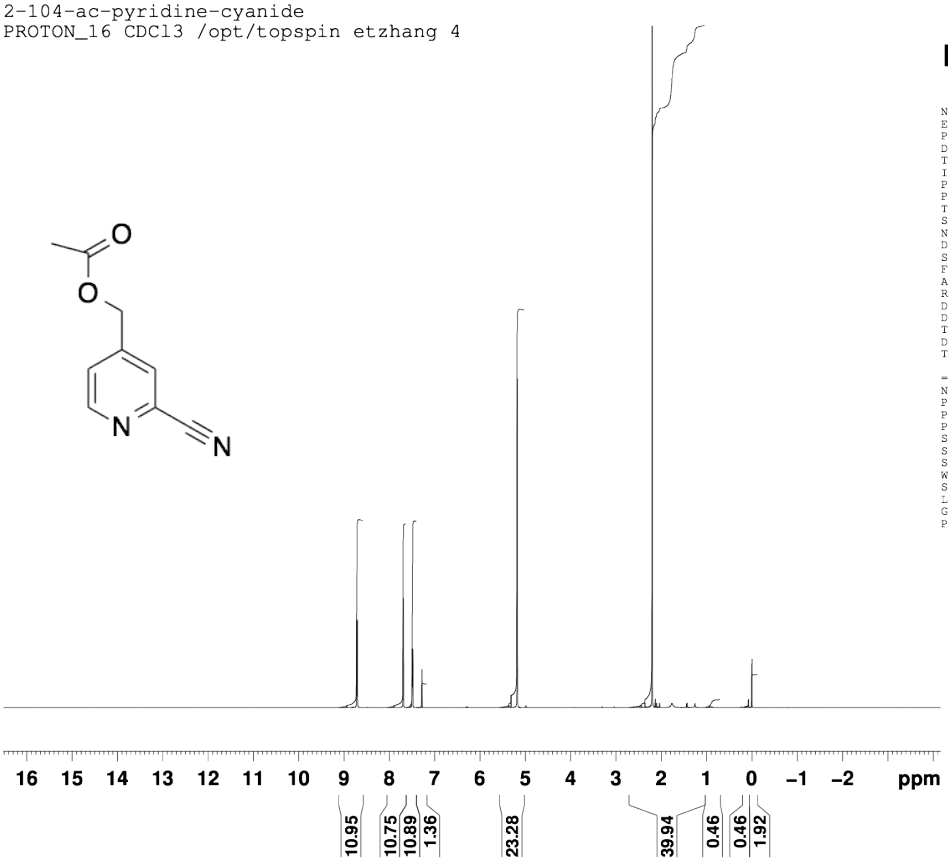

```
NAME          2-102-ac-pyridine-n-oxide
EXPNO                 1
PROCNO                1
Date_          20120905
Time               17.33
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                 11.3
DW               60.400 usec
DE                6.00 usec
TE               298.2 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                15.00 usec
PL1                4.90 dB
PL1W        3.30822015 W
SFO1       400.1324710 MHz
SI                32768
SF         400.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

2-102-ac-pyridine-n-oxide
C13CPD1024 CDCl3 /opt/topspin etzhang 3



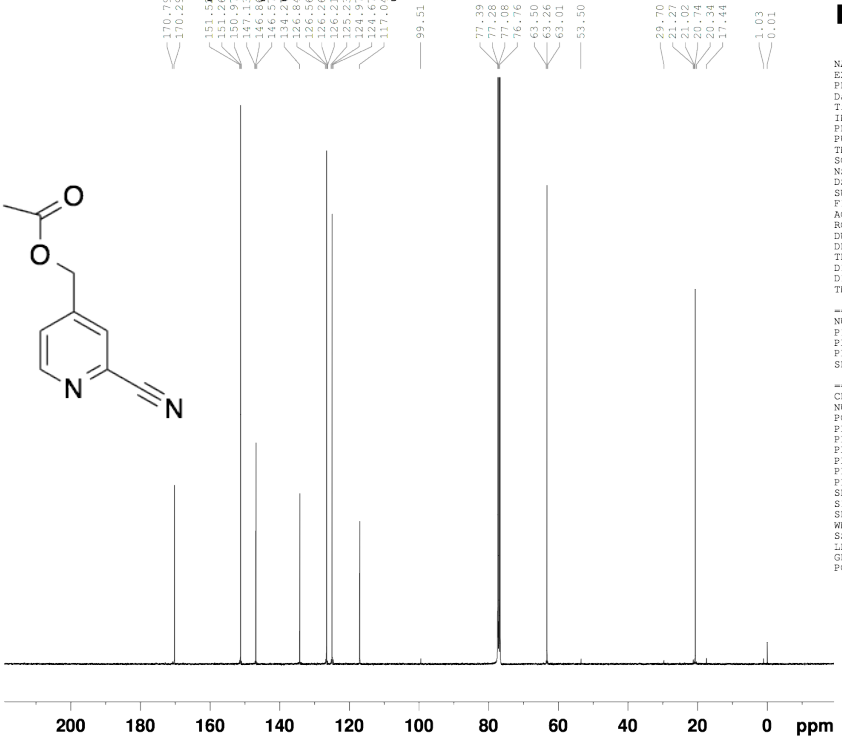

```
NAME          2-102-ac-pyridine-n-oxide
EXPNO                 3
PROCNO                1
Date_          20120906
Time               0.05
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH          23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                  512
DW               20.850 usec
DE                18.00 usec
TE               298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                9.25 usec
PL1                0.55 dB
PL1W       35.18820572 W
SFO1      100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2                4.90 dB
PL12              20.46 dB
PL13              21.00 dB
PL2W        3.30822015 W
PL12W       0.09195905 W
PL13W       0.08120718 W
SFO2      400.1316005 MHz
SI                32768
SF        100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

2-104-ac-pyridine-cyanide
PROTON_16 CDCl3 /opt/topspin etzhang 4



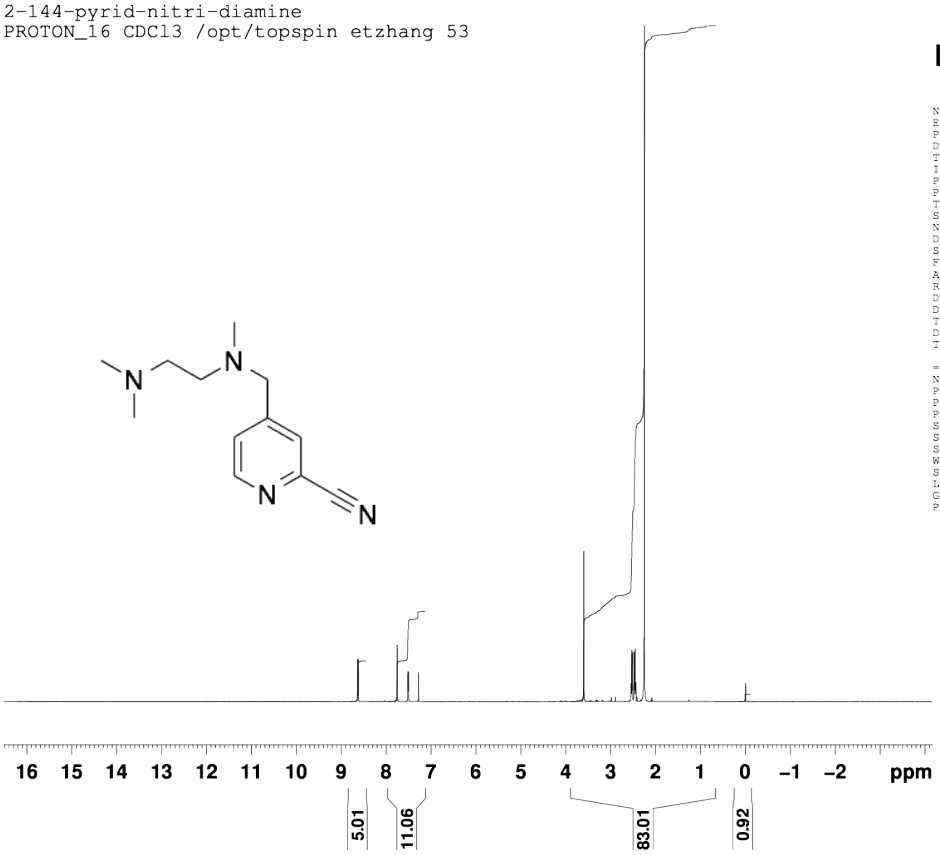

```
NAME          2-104-ac-pyridine-cyanide
EXPNO                 1
PROCNO                1
Date_          20120905
Time              17.40
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                11.3
DW              60.400 usec
DE               6.00 usec
TE              298.2 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                15.00 usec
PL1                4.90 dB
PL1W         3.30822015 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

2-104-ac-pyridine-cyanide
C13CPD1024 CDCl3 /opt/topspin etzhang 4



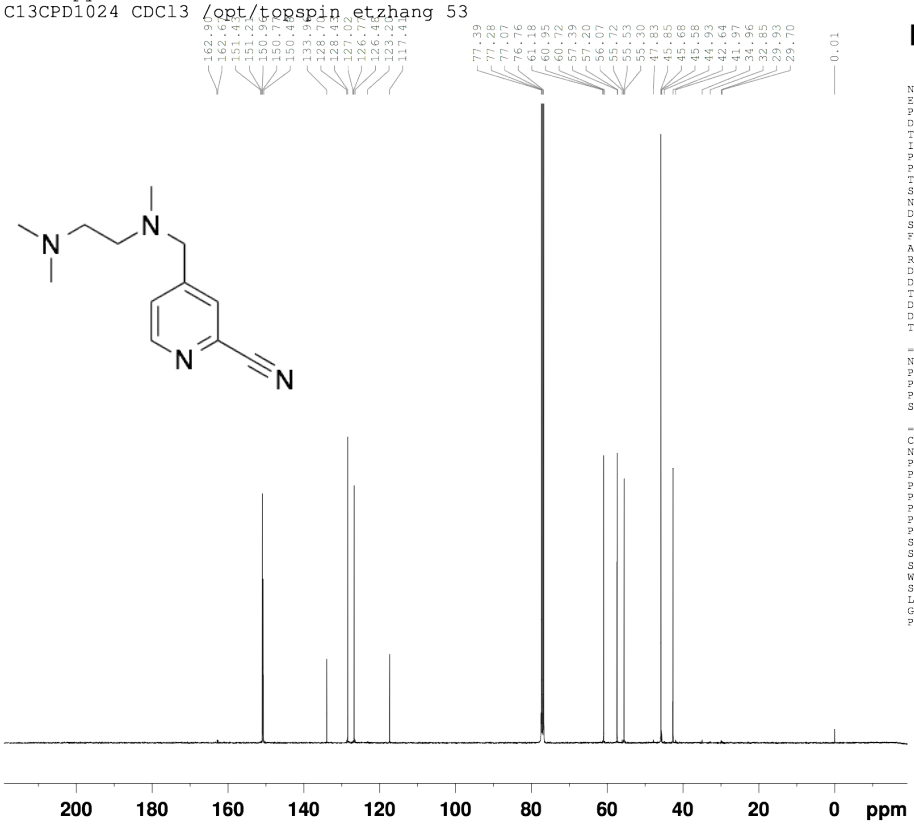

```
NAME          2-104-ac-pyridine-cyanide
EXPNO                 3
PROCNO                1
Date_          20120906
Time              1.24
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                1024
DS                   4
SWH           23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                 512
DW              20.850 usec
DE              18.00 usec
TE              298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                9.25 usec
PL1               0.55 dB
PL1W        35.18820572 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2               4.90 dB
PL12             20.46 dB
PL13             21.00 dB
PL2W         3.30822015 W
PL12W        0.09195905 W
PL13W        0.08120718 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

2-144-pyrid-nitri-diamine
PROTON_16 CDCl3 /opt/topspin etzhang 53



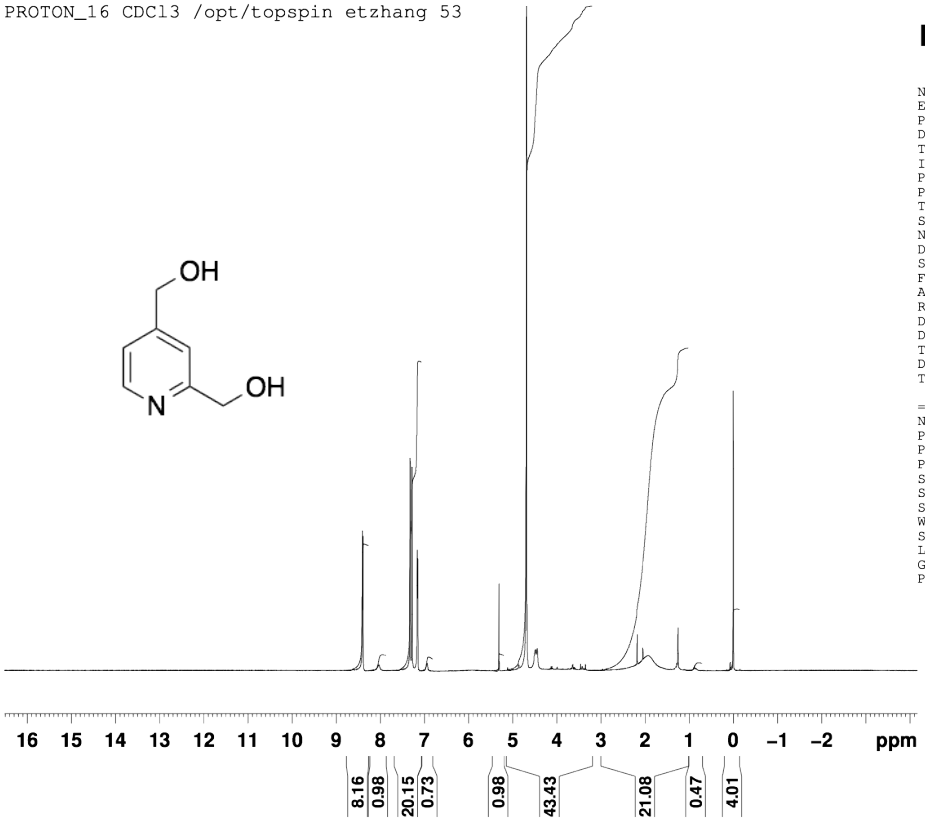

```
NAME        2-144-pyrid-nitri-diamine-
EXPNO                 1
PROCNO                1
Date_          20121121
Time              17.59
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   16
DS                    2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                   9
DW               60.400 usec
DE                6.00 usec
TE               298.1 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                15.00 usec
PL1                4.90 dB
PL1W         3.30822015 W
SFO1        400.1324710 MHz
SI                32768
SF          400.1300000 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

2-144-pyrid-nitri-diamine
C13CPD1024 CDCl3 /opt/topspin etzhang 53



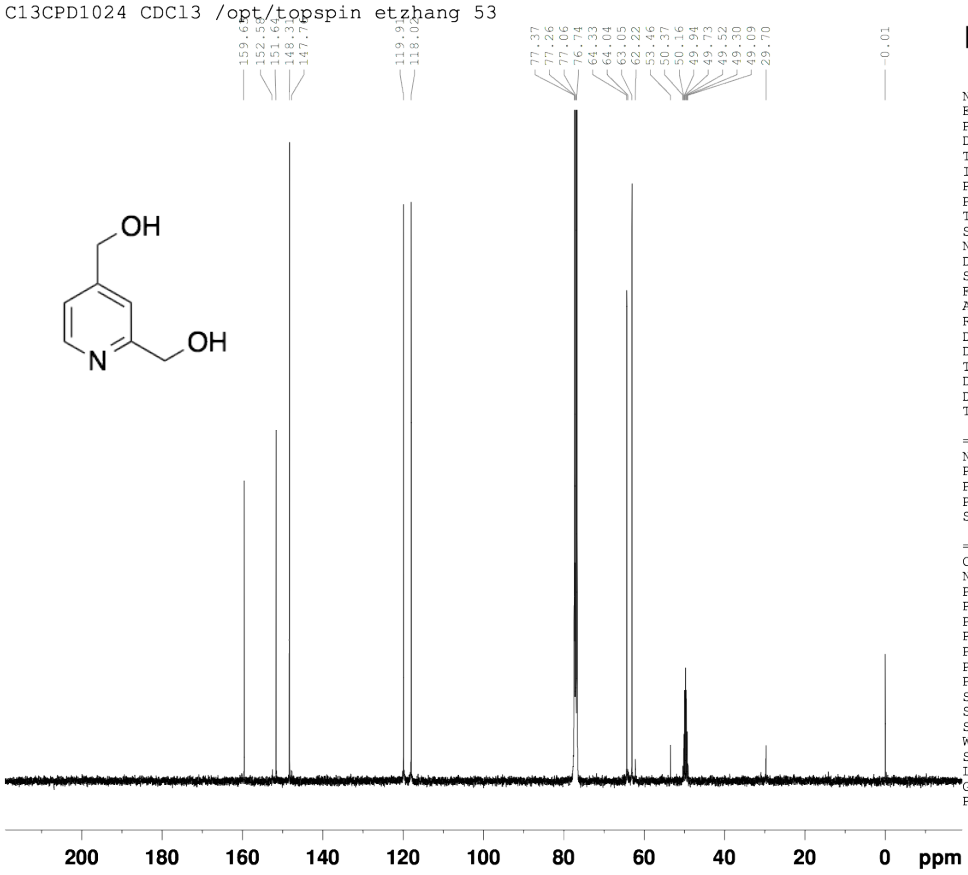

```
NAME        2-144-pyrid-nitri-diamine-
EXPNO                 3
PROCNO                1
Date_          20121122
Time              4.04
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                 1024
DS                    4
SWH          23980.814 Hz
FIDRES        0.365918 Hz
AQ            1.3664756 sec
RG                  512
DW               20.850 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                9.25 usec
PL1                0.55 dB
PL1W        35.18820572 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2                4.90 dB
PL12              20.46 dB
PL13              21.00 dB
PL2W         3.30822015 W
PL12W        0.09195905 W
PL13W        0.08120718 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127690 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

2-268
PROTON_16 CDCl3 /opt/topspin etzhang 53



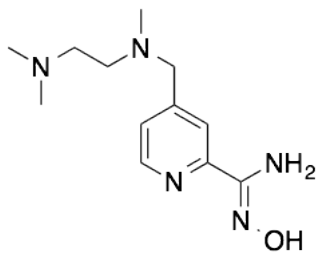
NAME	2-268
EXPNO	1
PROCNO	1
Date_	20130507
Time	17.51
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	9
DW	60.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

2-268
C13CPD1024 CDCl3 /opt/topspin etzhang 53



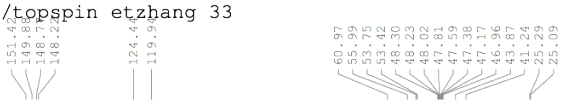
NAME	2-268
EXPNO	4
PROCNO	1
Date_	20130507
Time	21.04
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	512
DW	20.850 usec
DE	18.00 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.25 usec
PL1	0.55 dB
PL1W	35.18820572 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	4.90 dB
PL12	20.46 dB
PL13	21.00 dB
PL2W	3.30822015 W
PL12W	0.09195905 W
PL13W	0.08120718 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

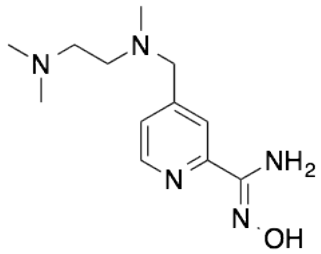
2-216
PROTON_16 MeOD /opt/topspin etzhang 33



NAME	2-216
EXPNO	1
PROCNO	1
Date_	20130321
Time	8.04
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	6.3
DW	60.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

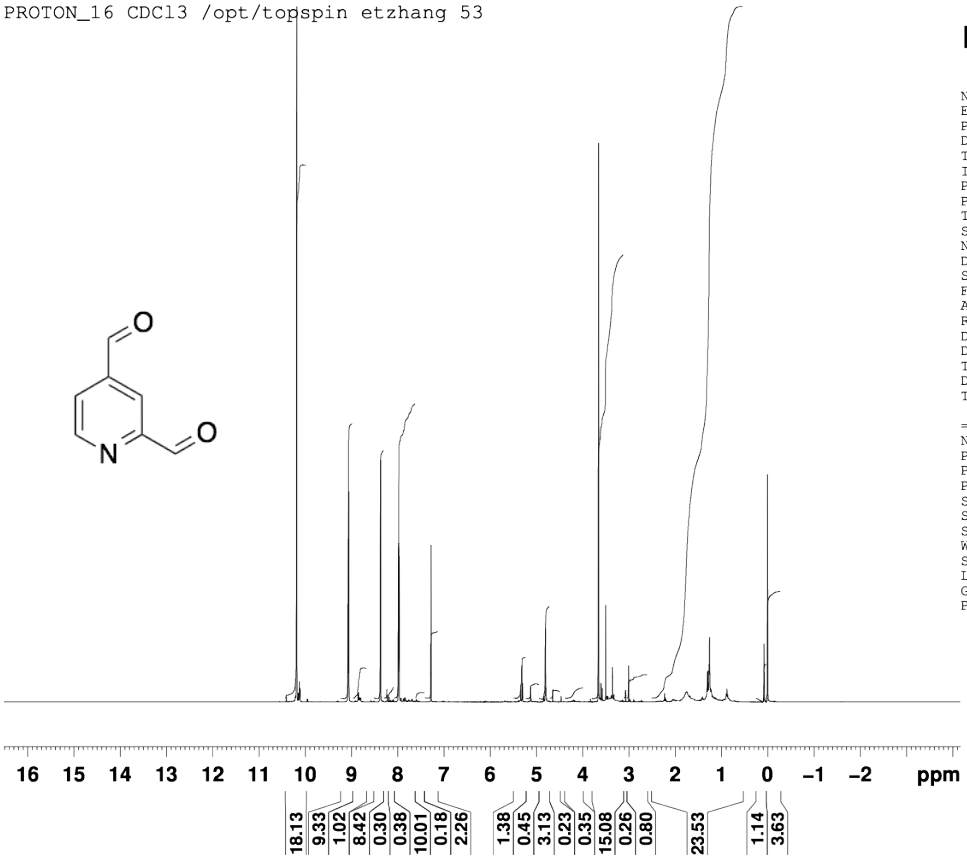
2-216
C13CPD1024 MeOD /opt/topspin etzhang 33





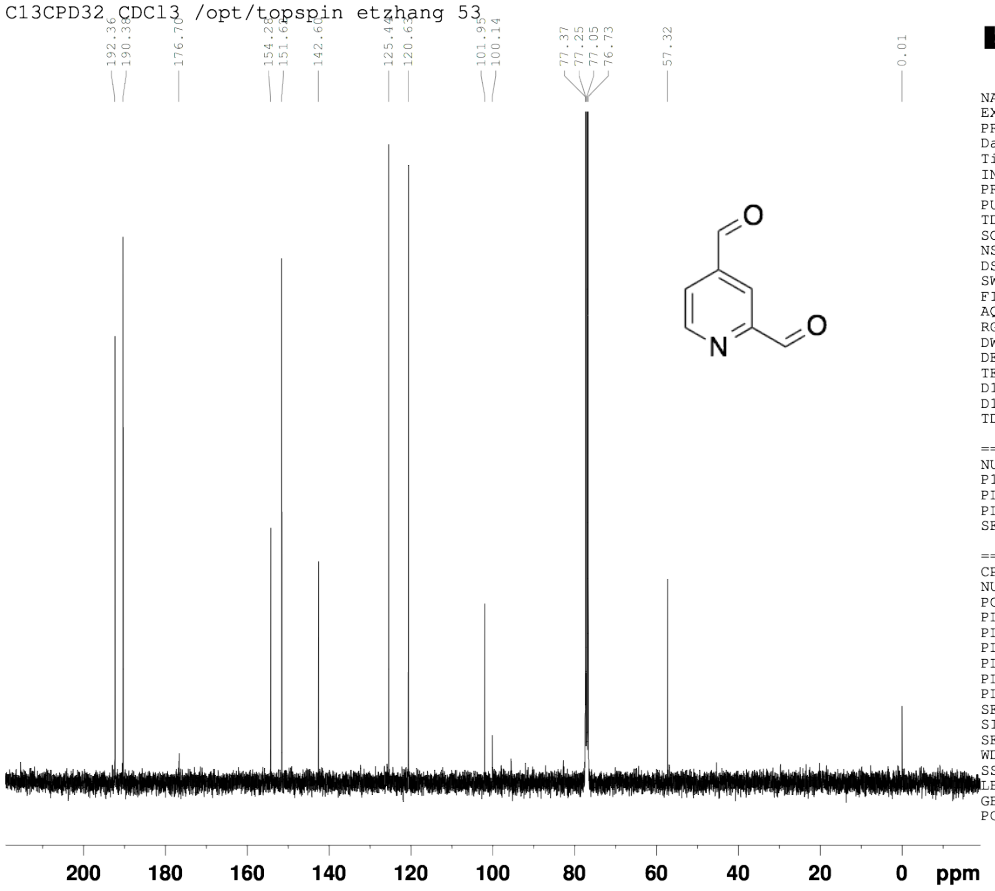
NAME	2-216
EXPNO	4
PROCNO	1
Date_	20130322
Time	6.04
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	MeOD
NS	1024
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	512
DW	20.850 usec
DE	18.00 usec
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.25 usec
PL1	0.55 dB
PL1W	35.18820572 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	4.90 dB
PL12	20.46 dB
PL13	21.00 dB
PL2W	3.30822015 W
PL12W	0.09195905 W
PL13W	0.08120718 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

2-271
PROTON_16 CDCl3 /opt/topspin etzhang 53



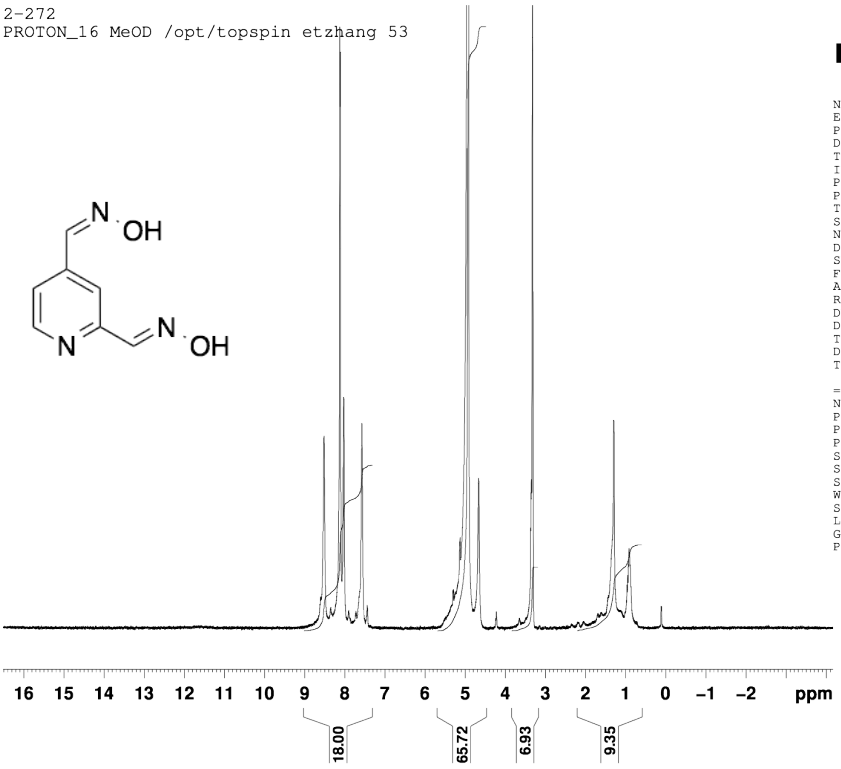
NAME	2-271
EXPNO	1
PROCNO	1
Date_	20130508
Time	18.31
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	9
DW	60.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

2-271
C13CPD32 CDCl3 /opt/topspin etzhang 53



NAME	2-271
EXPNO	3
PROCNO	1
Date_	20130508
Time	18.47
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	512
DW	20.850 usec
DE	18.00 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.25 usec
PL1	0.55 dB
PL1W	35.18820572 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	4.90 dB
PL12	20.46 dB
PL13	21.00 dB
PL2W	3.30822015 W
PL12W	0.09195905 W
PL13W	0.08120718 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

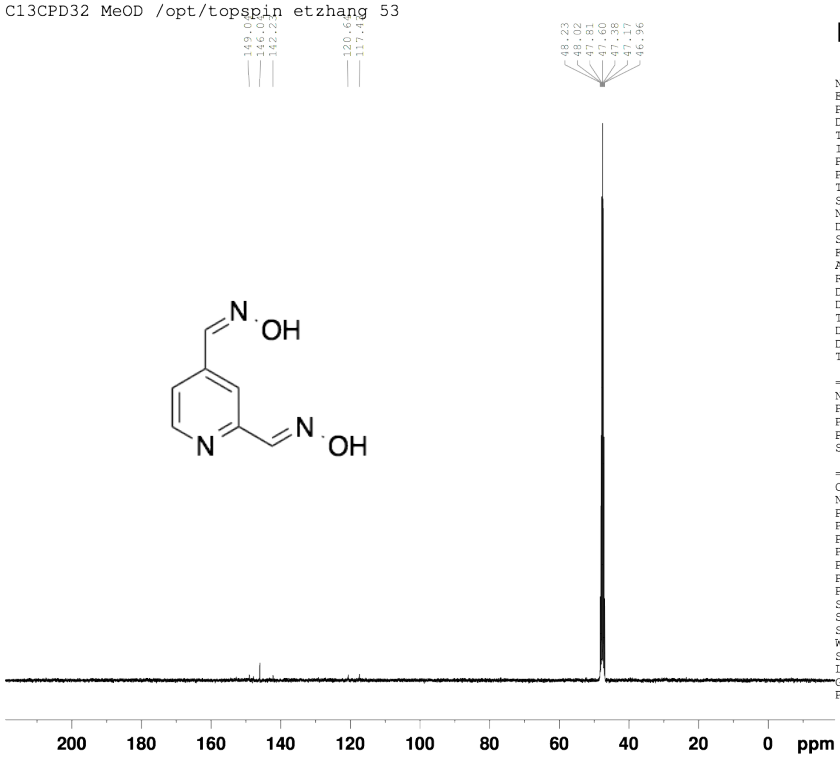
2-272
PROTON_16 MeOD /opt/topspin etzhang 53



BRUKER

```
NAME                  2-272
EXPNO                     1
PROCNO                    1
Date_              20130510
Time                   5.44
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG                zg30
TD                    65536
SOLVENT                MeOD
NS                       16
DS                        2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                        4
DW                   60.400 usec
DE                    6.00 usec
TE                   298.2 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                   15.00 usec
PL1                   4.90 dB
PL1W            3.30822015 W
SFO1          400.1324710 MHz
SI                    32768
SF            400.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

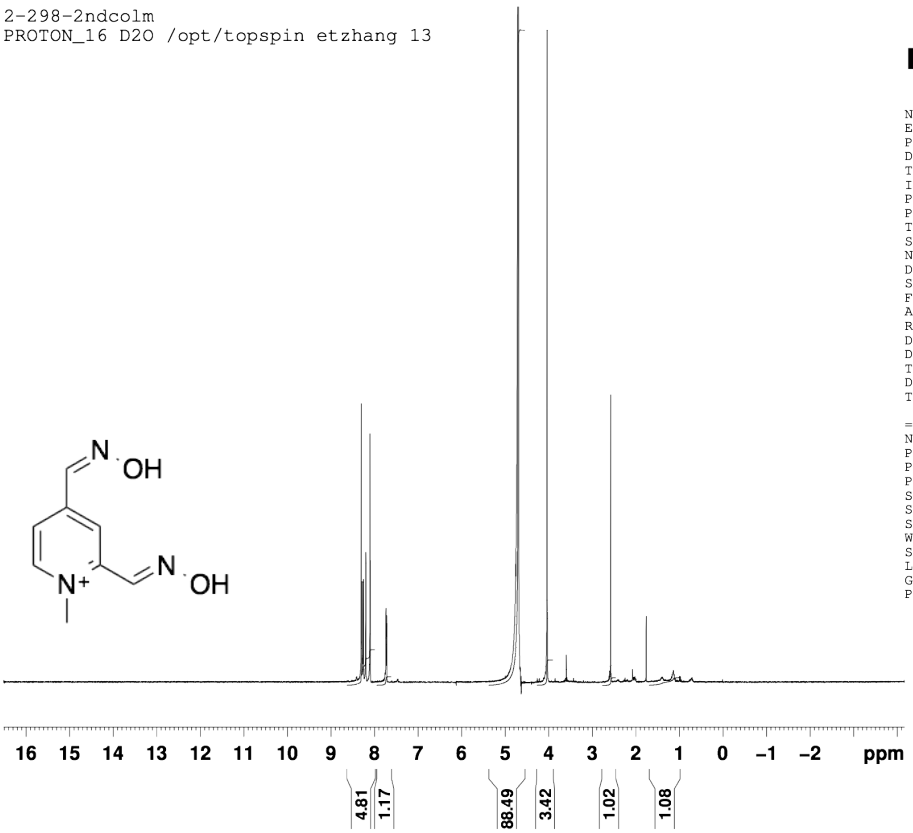
2-272
C13CPD32 MeOD /opt/topspin etzhang 53



BRUKER

```
NAME                  2-272
EXPNO                     3
PROCNO                    1
Date_              20130510
Time                   5.59
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zgpg30
TD                    65536
SOLVENT                MeOD
NS                       32
DS                        4
SWH              23980.814 Hz
FIDRES            0.365918 Hz
AQ                1.3664756 sec
RG                      512
DW                   20.850 usec
DE                    18.00 usec
TE                   298.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.25 usec
PL1                   0.55 dB
PL1W           35.18820572 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                   4.90 dB
PL12                 20.46 dB
PL13                 21.00 dB
PL2W            3.30822015 W
PL12W           0.09195905 W
PL13W           0.08120718 W
SFO2          400.1316005 MHz
SI                    32768
SF            100.6127690 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

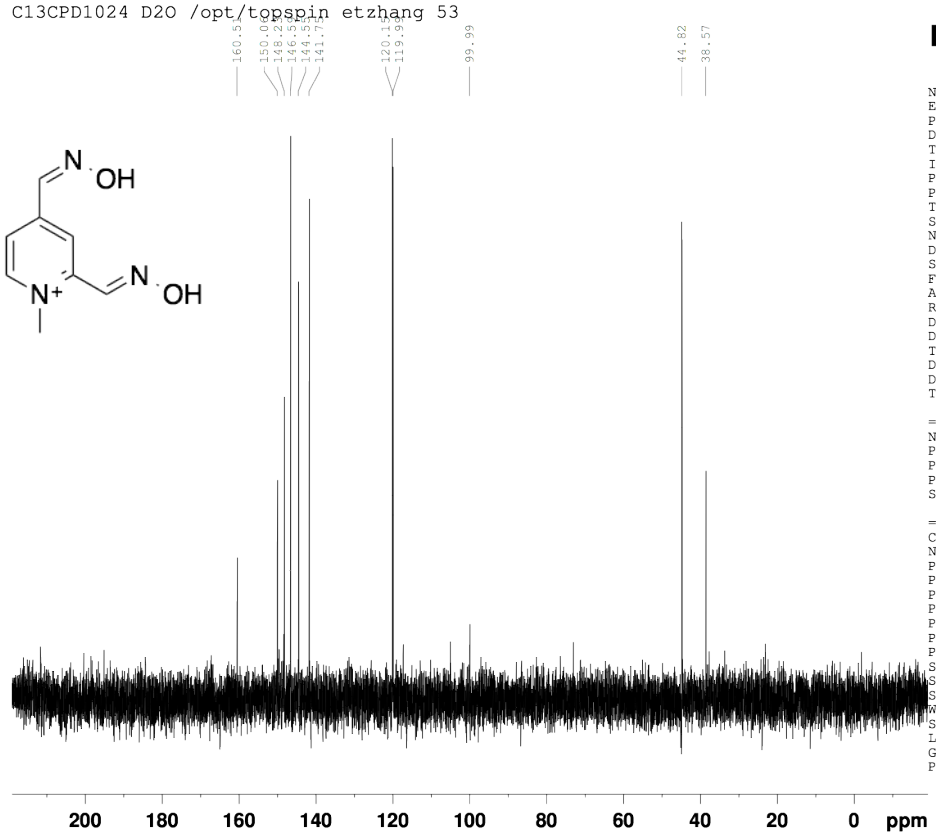
2-298-2ndcolm
PROTON_16 D2O /opt/topspin etzhang 13



BRUKER

NAME	2-298-2ndcolm
EXPNO	1
PROCNO	1
Date_	20130606
Time	10.24
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zg30
TD	65536
SOLVENT	D2O
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	9
DW	60.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

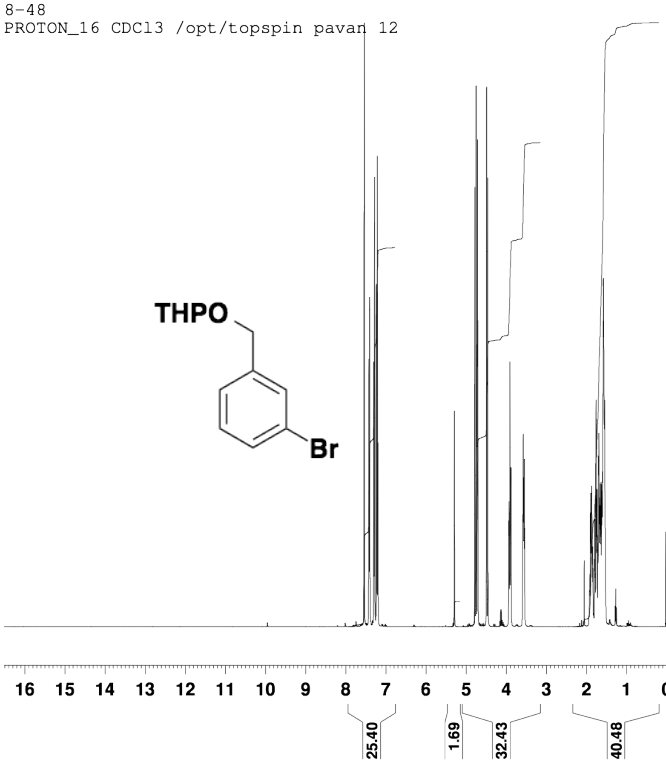
2-298-2nd-colm
C13CPD1024 D2O /opt/topspin etzhang 53



BRUKER

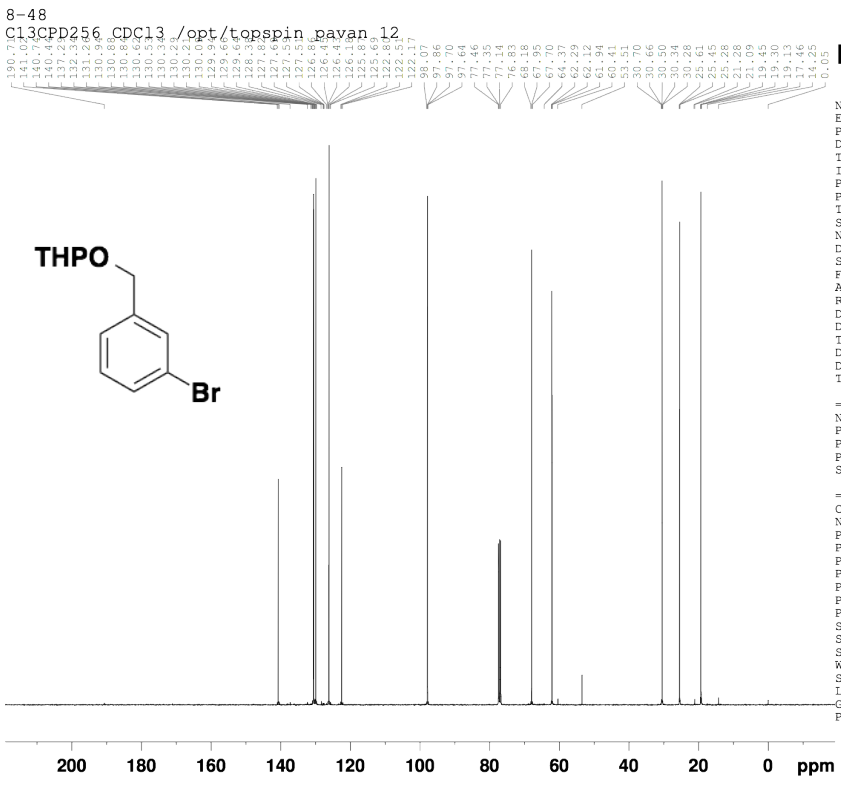
NAME	2-298-2ndcolm
EXPNO	4
PROCNO	1
Date_	20130608
Time	4.40
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	D2O
NS	1024
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	512
DW	20.850 usec
DE	18.00 usec
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.25 usec
PL1	0.55 dB
PL1W	35.18820572 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	4.90 dB
PL12	20.46 dB
PL13	21.00 dB
PL2W	3.30822015 W
PL12W	0.09195905 W
PL13W	0.08120718 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

8-48
PROTON_16 CDCl3 /opt/topspin pavan 12



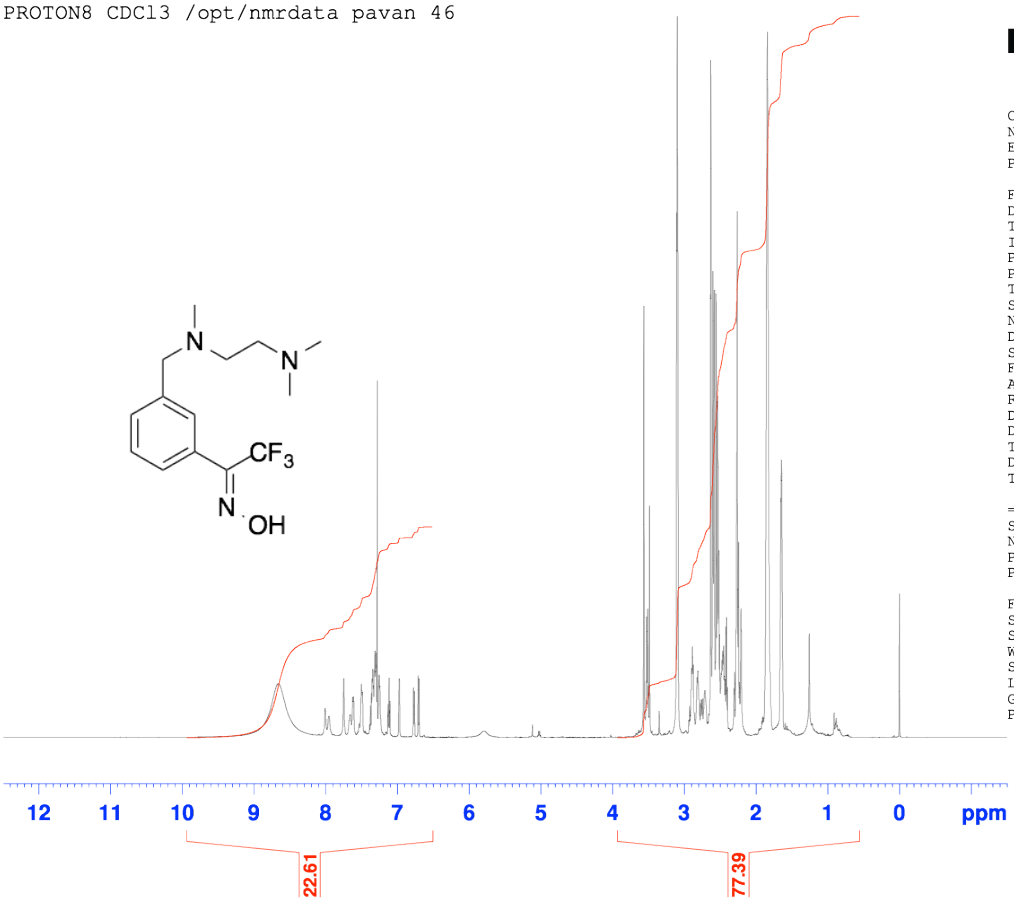

```
NAME                Nov29-2012
EXPNO                        1
PROCNO                       1
Date_                 20121130
Time                      3.28
INSTRUM                  spect
PROBHD    5 mm CPQNP 1H/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8278.146 Hz
FIDRES               0.126314 Hz
AQ                   3.9584243 sec
RG                          8
DW                     60.400 usec
DE                      6.00 usec
TE                     298.1 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                      15.00 usec
PL1                      4.90 dB
PL1W                3.30822015 W
SFO1              400.1324710 MHz
SI                        32768
SF                400.1300000 MHz
WDW                          EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                         1.00
```

8-48
C13CPD256 CDCl3 /opt/topspin pavan 12



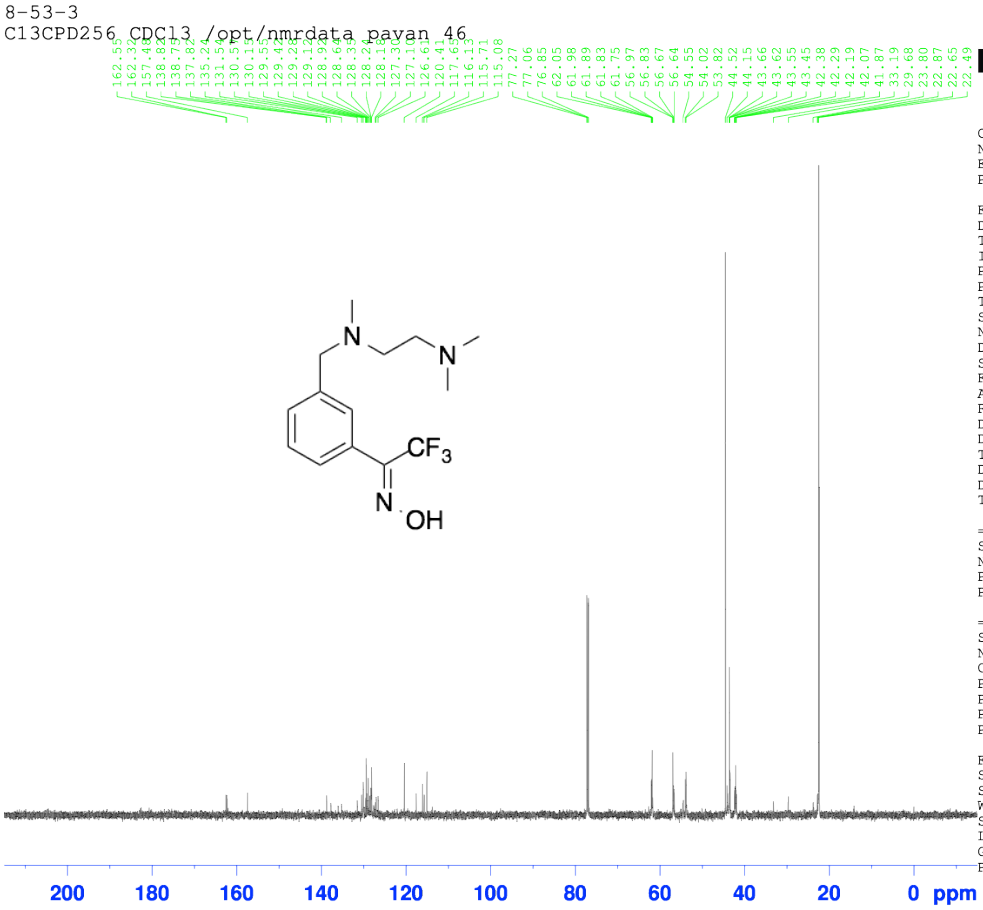

```
NAME                Nov29-2012
EXPNO                        2
PROCNO                       1
Date_                 20121130
Time                      3.44
INSTRUM                  spect
PROBHD    5 mm CPQNP 1H/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         256
DS                          4
SWH                 23980.814 Hz
FIDRES               0.365918 Hz
AQ                   1.3664756 sec
RG                        512
DW                     20.850 usec
DE                      18.00 usec
TE                     298.2 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                       9.25 usec
PL1                      0.55 dB
PL1W               35.18820572 W
SFO1              100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                   90.00 usec
PL2                      4.90 dB
PL12                    20.46 dB
PL13                    21.00 dB
PL2W                3.30822015 W
PL12W               0.09195905 W
PL13W               0.08120718 W
SFO2              400.1316005 MHz
SI                        32768
SF                100.6127690 MHz
WDW                          EM
SSB                           0
LB                       1.00 Hz
GB                            0
PC                         1.40
```

8-53-3
PROTON8 CDCl3 /opt/nmrdata pavan 46



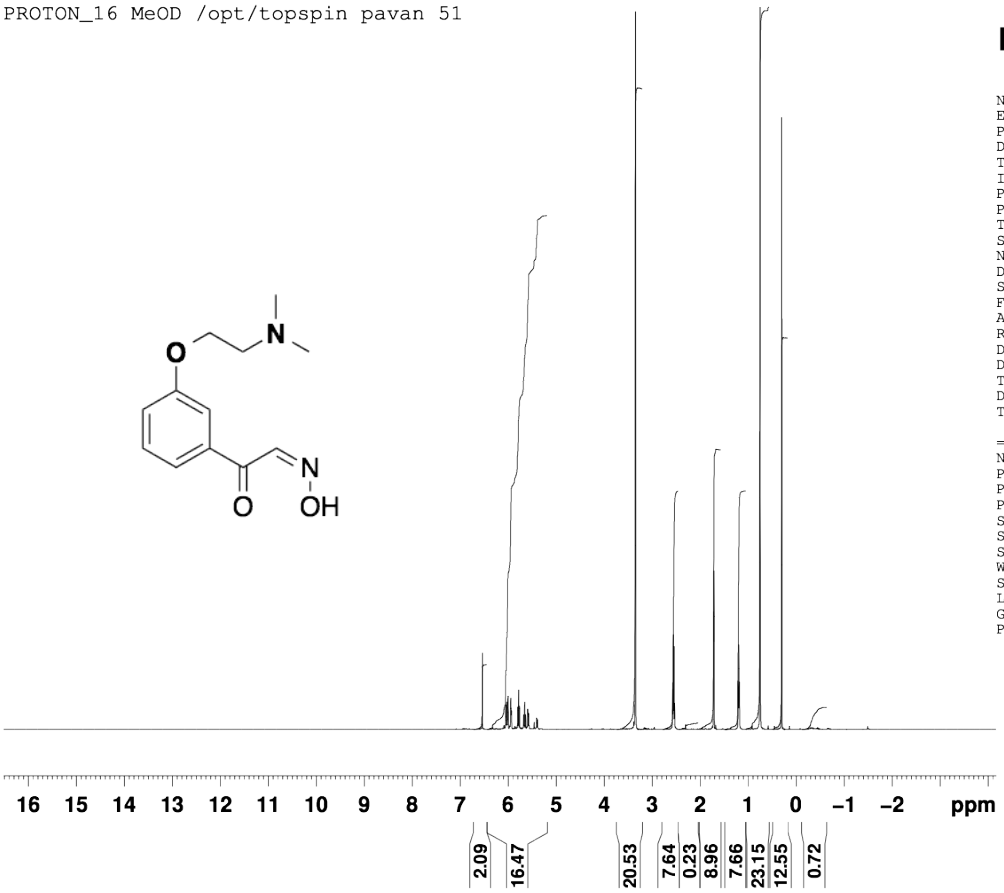

```
Current Data Parameters
NAME          Dec12-2012
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121212
Time               21.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    2
SWH            8403.361 Hz
FIDRES         0.128225 Hz
AQ             3.8993919 sec
RG                   57
DW               59.500 usec
DE               17.39 usec
TE                298.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.3233018 MHz
NUC1                   1H
P1                10.77 usec
PLW1         26.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.3200022 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

8-53-3
C13CPD256 CDCl3 /opt/nmrdata pavan 46



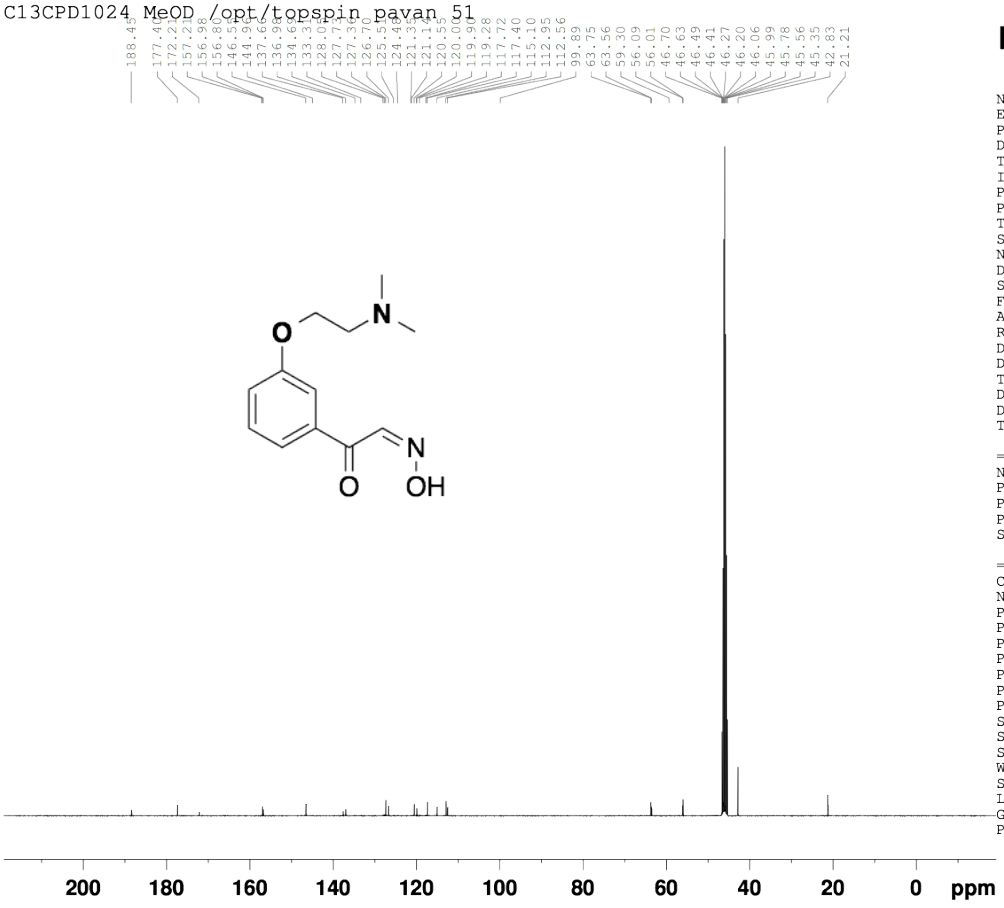

```
Current Data Parameters
NAME          Dec12-2012
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121212
Time               21.30
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg55
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           34722.223 Hz
FIDRES         0.529819 Hz
AQ             0.9437184 sec
RG                 2050
DW               14.400 usec
DE               19.34 usec
TE                298.1 K
D1            1.10000002 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9656784 MHz
NUC1                  13C
P1                10.63 usec
PLW1        110.00000000 W

======== CHANNEL f2 ========
SFO2         600.3224013 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2             70.00 usec
PLW2         22.00000000 W
PLW12         0.51885003 W
PLW13         0.25424001 W

F2 - Processing parameters
SI                 32768
SF           150.9505840 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

8-86
PROTON_16 MeOD /opt/topspin pavan 51



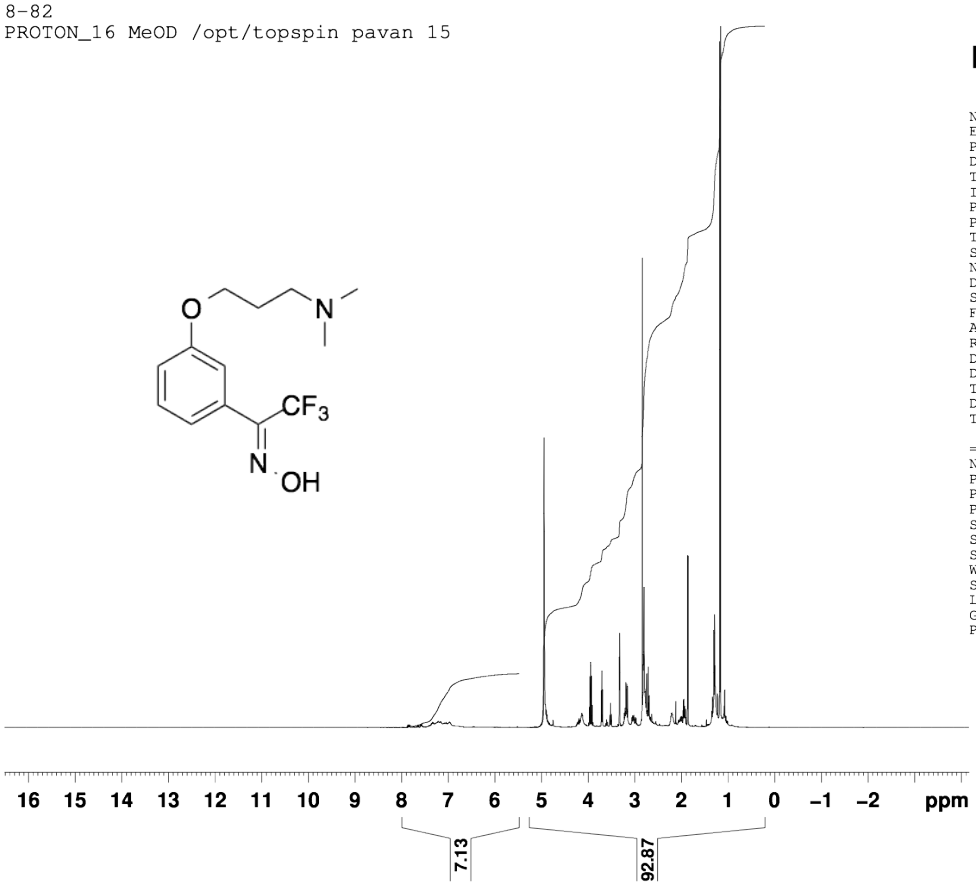
NAME	Apr13-2013
EXPNO	1
PROCNO	1
Date_	20130414
Time	8.19
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	32
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	4
DW	60.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

8-86
C13CPD1024 MeOD /opt/topspin pavan 51



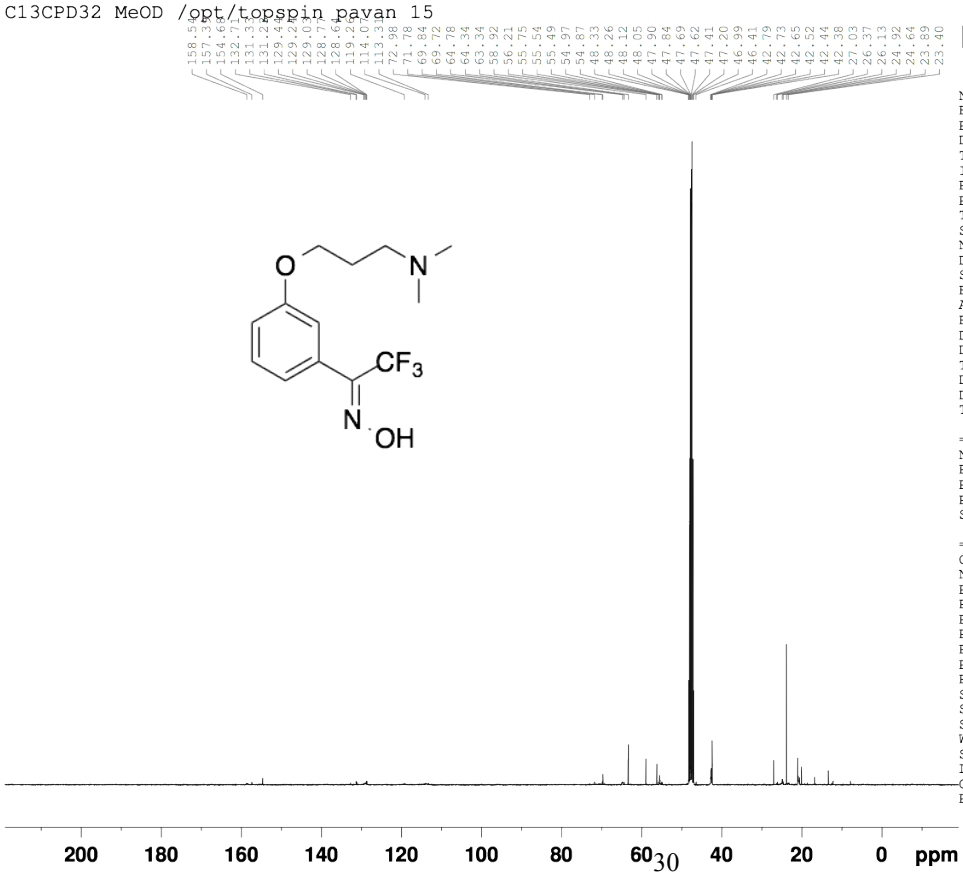
NAME	Apr13-2013
EXPNO	2
PROCNO	1
Date_	20130414
Time	9.19
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	MeOD
NS	1024
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	512
DW	20.850 usec
DE	18.00 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.25 usec
PL1	0.55 dB
PL1W	35.18820572 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	4.90 dB
PL12	20.46 dB
PL13	21.00 dB
PL2W	3.30822015 W
PL12W	0.09195905 W
PL13W	0.08120718 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

8-82
PROTON_16 MeOD /opt/topspin pavan 15



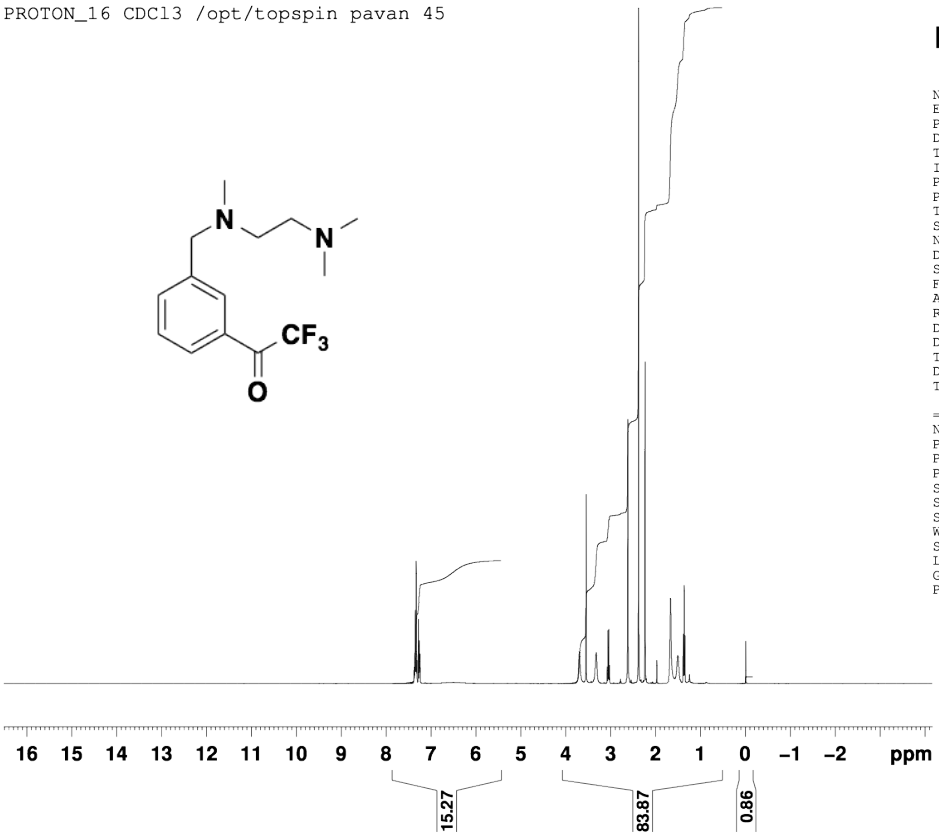
NAME	Apr01-2013
EXPNO	1
PROCNO	1
Date_	20130401
Time	15.08
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zg30
TD	65536
SOLVENT	MeOD
NS	32
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	4
DW	60.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

8-82
C13CPD32 MeOD /opt/topspin pavan 15



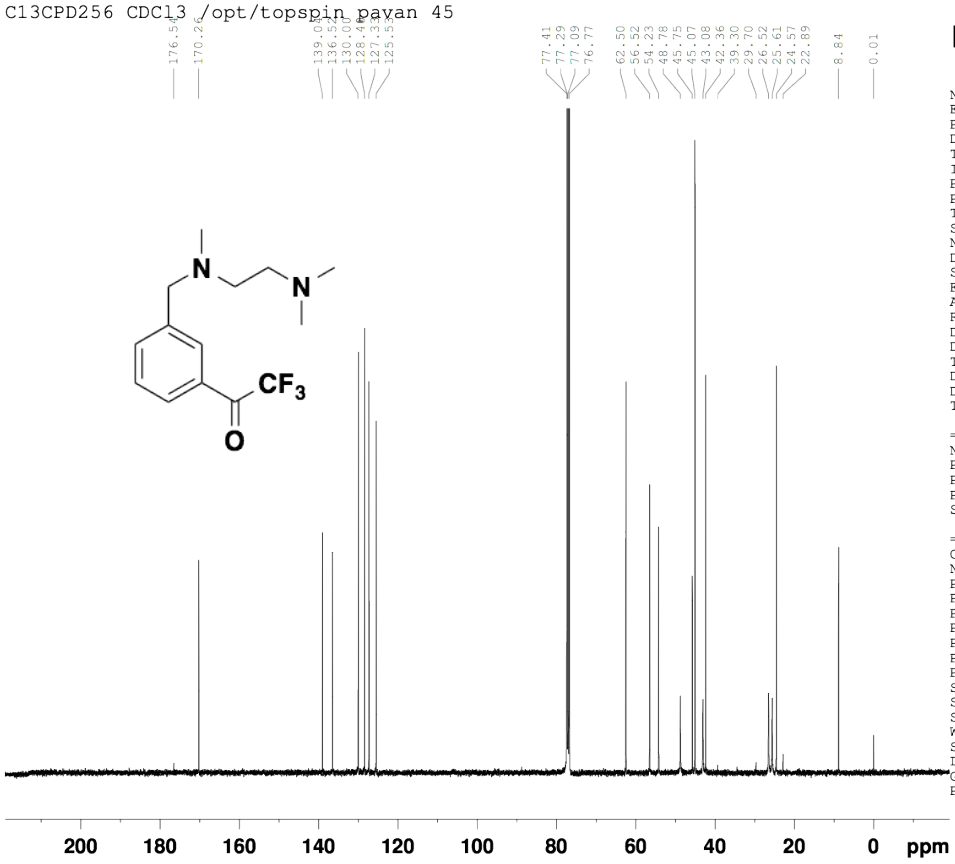
NAME	Apr01-2013
EXPNO	2
PROCNO	1
Date_	20130401
Time	15.24
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	MeOD
NS	256
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	512
DW	20.850 usec
DE	18.00 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.25 usec
PL1	0.55 dB
PL1W	35.18820572 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	4.90 dB
PL12	20.46 dB
PL13	21.00 dB
PL2W	3.30822015 W
PL12W	0.09195905 W
PL13W	0.08120718 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

8-52
PROTON_16 CDCl3 /opt/topspin pavan 45



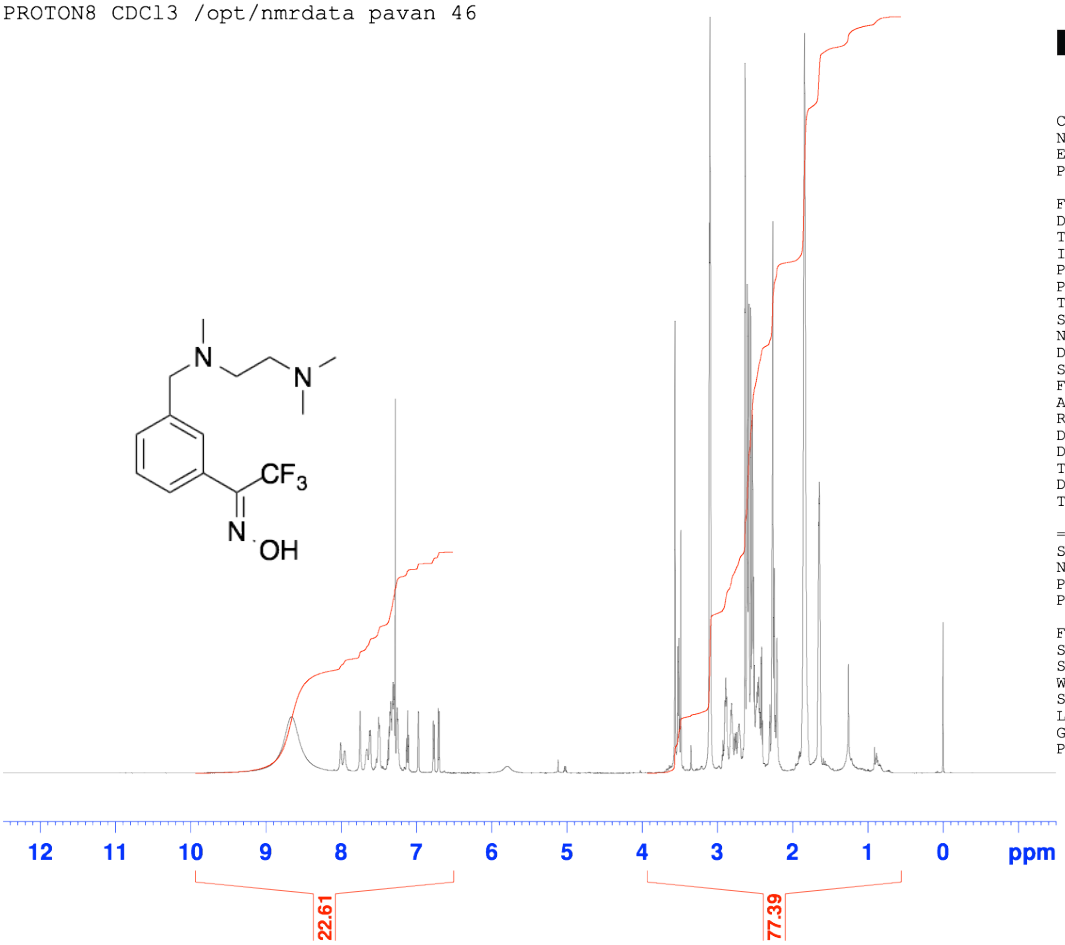
NAME	Dec10-2012
EXPNO	1
PROCNO	1
Date_	20121211
Time	7.55
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	11.3
DW	60.400 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.00 usec
PL1	4.90 dB
PL1W	3.30822015 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

8-52
C13CPD256 CDCl3 /opt/topspin pavan 45



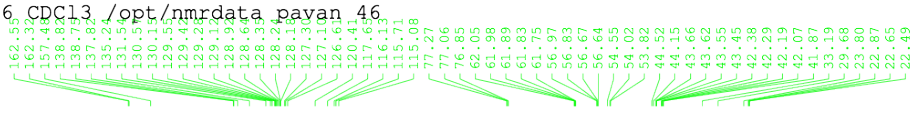
NAME	Dec10-2012
EXPNO	2
PROCNO	1
Date_	20121211
Time	9.04
INSTRUM	spect
PROBHD	5 mm CPQNP 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	23980.814 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	512
DW	20.850 usec
DE	18.00 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.25 usec
PL1	0.55 dB
PL1W	35.18820572 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	4.90 dB
PL12	20.46 dB
PL13	21.00 dB
PL2W	3.30822015 W
PL12W	0.09195905 W
PL13W	0.08120718 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

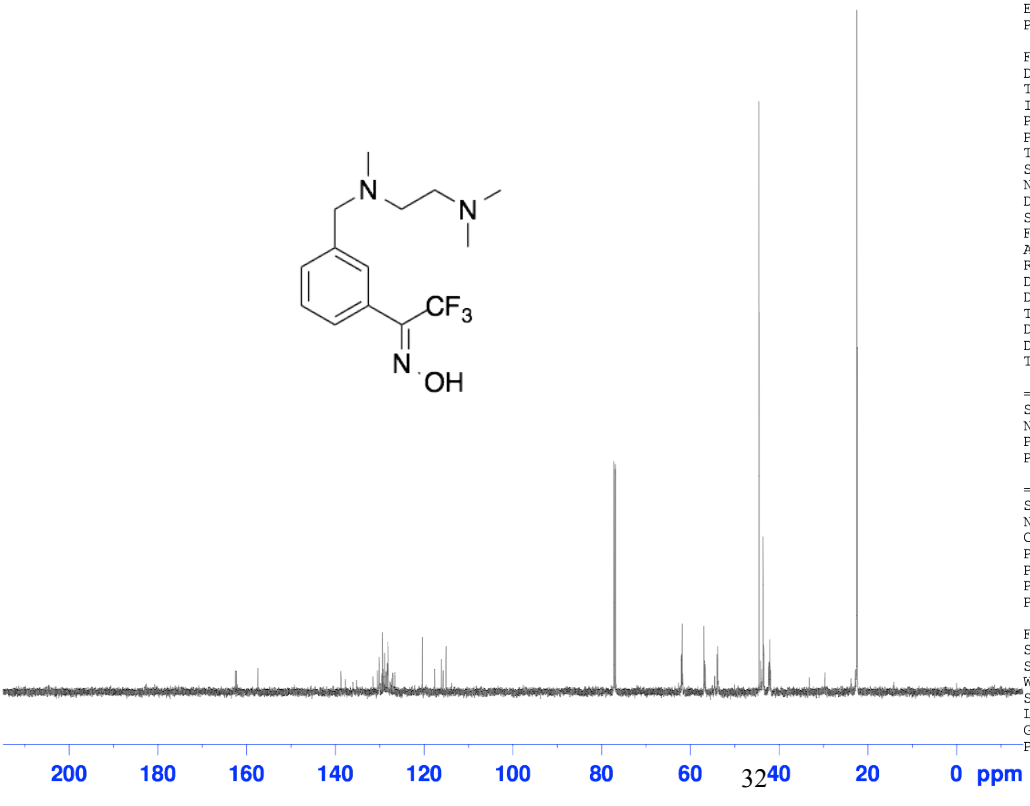
8-53-3
PROTON8 CDCl3 /opt/nmrdata pavan 46




```
Current Data Parameters
NAME          Dec12-2012
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121212
Time               21.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     2
SWH             8403.361 Hz
FIDRES          0.128225 Hz
AQ              3.8993919 sec
RG                    57
DW                59.500 usec
DE                17.39 usec
TE                298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.3233018 MHz
NUC1                   1H
P1                 10.77 usec
PLW1         26.00000000 W

F2 - Processing parameters
SI                 65536
SF          600.3200022 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

8-53-3
C13CPD256 CDCl3 /opt/nmrdata pavan 46






```
Current Data Parameters
NAME          Dec12-2012
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121212
Time               21.30
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg55
TD                 65536
SOLVENT            CDCl3
NS                   256
DS                     4
SWH            34722.223 Hz
FIDRES          0.529819 Hz
AQ              0.9437184 sec
RG                  2050
DW                14.400 usec
DE                19.34 usec
TE                298.1 K
D1             1.10000002 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9656784 MHz
NUC1                   13C
P1                 10.63 usec
PLW1        110.00000000 W

======== CHANNEL f2 ========
SFO2         600.3224013 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              70.00 usec
PLW2         22.00000000 W
PLW12         0.51885003 W
PLW13         0.25424001 W

F2 - Processing parameters
SI                 32768
SF          150.9505840 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```